A Chilling Embrace: Workholding With Ice

by

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Abstract:

This thesis explores the usage of ice as a fixturing medium in CNC machining of fragile components. Ice is used both as an adhesive and a potting compound to hold and support work during cutting. Measurements of ice adhesion strength as a function of workpiece material, roughness, and surface preparation are taken and analyzed. The research was carried out on a custom freeze chucking apparatus which was designed and constructed as part of the research. The results of the work show that ice can have substantial holding power, greatly exceeding machining forces, given that surface preparation is carried out in the prescribed manner.

Acknowledgements

I would like to thank my supervisor, Dr. Andrew Warkentin, for his guidance through this project. I would also like to thank my mentors, Ian Padden and Paul Moir, for teaching me most everything I know about engineering and design and being far too generous with their time and expertise. Without their guidance, this project could never have happened.

Essa Horvath has been a most diligent editor and friend through writing of this thesis, and credit for my continued sanity rests squarely on her shoulders. My family has always been the foundation on which I have approached my work, and their support in all my endeavors has been instrumental to everything I have ever achieved.

Chapter 1 - Introduction

As engineering has become more advanced, and our ability to optimize parameters and designs with simulation has increased, more and more parts are being designed which require exotic machining strategies. Along with exotic machining strategies, many parts require exotic fixturing strategies to account for fragile materials which cannot undergo the stress of being mechanically clamped or parts which might be made too fragile for mechanical clamps during the course of machining. Introduction of new materials such as ceramics, super alloys, and composites into large sector projects has pushed the development of both cutter and machine tool technology. Development of highly engineered parts using simulation, optimized ribbed structures, and micro-scale features have also forced advancements in holding the parts while they're being machined. Machining ceramics, glasses, and other brittle materials or parts with fragile features rules out using strictly mechanical clamping, and so modern machine shops use techniques including vacuum clamping, adhesives, and potting (encasing parts) in low melting or soluble compounds for cutting.

One of the more novel solutions to the problem of holding small and fragile parts, and the topic of this work, is to use ice both as an adhesive and a potting compound. Ice can have a strong adhesive force, as evidenced by the adhesion of thousands of car doors and windshield wipers to car bodies and windscreens across Canada on a typical February morning. Additionally, ice is hard in the solid phase and can be used as a support material. Ice is readily available, non-toxic, and easy to clean. Freezing can take less than a minute, similarly for thawing, requiring only a simple change in temperature.

Research Goals:

While there are commercial devices available which take advantage of ice for workpiece fixturing(Horst Witte, 2010), the expense of the devices and the specialized nature of the work best utilizing them has resulted in little discussion or exploration into the specifics of their operation. There is little literature in the academic community specifically addressing this usage of ice as a fixturing medium, and its relevant properties.

The goal of this research is to provide a set of measured operating parameters such that a framework for evaluating this method versus others and a list of best practices in its use can be constructed. Specific parameters examined include the effects of workpiece material, surface preparation of workpiece and fixture, and surface condition of workpiece. This data can be used to determine both the effort required to implement the method and the expected effectiveness.

Outline of Thesis:

- Chapter 2 contains a review of fixturing in the field of machining. Specific attention is given to the applications of each type of fixturing in context, and an effort is made to be thorough with coverage of each of them.
- Chapter 3 is a review of adhesion and includes coverage of both the mechanics of adhesion and properties of different adhesives.
- Chapter 4 contains a review of current literature from both professional and academic sources on the relevant properties of ice as an adhesive.
- Chapter 5 contains a description of the design of the test apparatus and a review of the technologies used. The results of tests performed to determine the basic operating parameters and limits of the device are presented in this chapter.
- Chapter 6 establishes the testing methodology and variables to be measured, and discusses issues encountered during testing. It also includes the results of testing, and analysis of the data with respect to real world concerns with suggestions for future research in this area.

Chapter 2 – Quick Fixes : Focus on Fixtures in Fewer Than Forty Paragraphs

Fixturing is often the most difficult process in manufacturing complex parts. Computer control has allowed tools to execute complex motions with extreme precision, completely removing operator error from the equation when cutting or forming components. The repeatability of modern tools allows any errors caused by deflection of cutters or materials to be programmed out after test pieces are run. The software used for creation of machine tool and robot motion usually catches process errors before they hit the shop floor through a combination of cut simulation and reliable automatic motion generation algorithms.

While there is an art and nuance to designing cutting strategies and tool paths for milling and turning machinery, it is usually a matter of optimizing material removal and surface finish. Fixturing, on the other hand, can involve a large set of factors being balanced to provide sufficient hold down of a complex form to resist machining forces while ensuring that the holding force itself does not distort or damage the item being held. On top of balancing these forces, the fixturing solution must be designed in such a way that the cutting or forming tools can access all required areas of the object being machined while avoiding collision with the fixture.

This chapter will initially focus on an overview of fixturing methods used in modern manufacturing facilities with discussion of benefits and caveats involved with each. As it is one of the most demanding manufacturing processes in terms of forces exerted on workpieces and workpiece complexity, machining will be used as the basis on which different fixturing techniques are compared.

Clamping methods can be roughly divided into four categories: mechanical, adhesive, physical, and compound. Mechanical clamping methods hold work by use of simple machines such as screws and wedges, adhesive clamping methods adhere the workpiece to the fixture, physical process fixturing uses physical properties such as vacuum or thermal contraction to hold workpieces, and compound fixturing systems fixture a workpiece by making it part of a larger workpiece which is easier to fixture via some other method.

2.1 Mechanical Clamping:

Mechanical clamping processes all work on the general principle of applying a force to push together a part and fixture such that friction holds them rigidly in relative position. The simplest mechanical clamp is a heavy weight placed onto the workpiece. Mechanical fixtures can also make use of interference such as, for example, putting a square workpiece into a square hole to prevent the workpiece from rotating while being processed. The defining factor of mechanical clamping systems besides their method of force generation is that generally the clamping apparatus can be removed and reused on another workpiece.

Probably the first mechanical clamp used in production was based on the simplest of the basic machines, the inclined plane or 'wedge'. Wedge clamping, shown in Figure 1, has a long history in woodworking, far before the invention of the screw or even the nail. All wedge clamping systems are essentially the same, involving a set of stops on both sides of a workpiece with the wedges forced between one side of the workpiece and one of the stops to force the workpiece against the other stop.

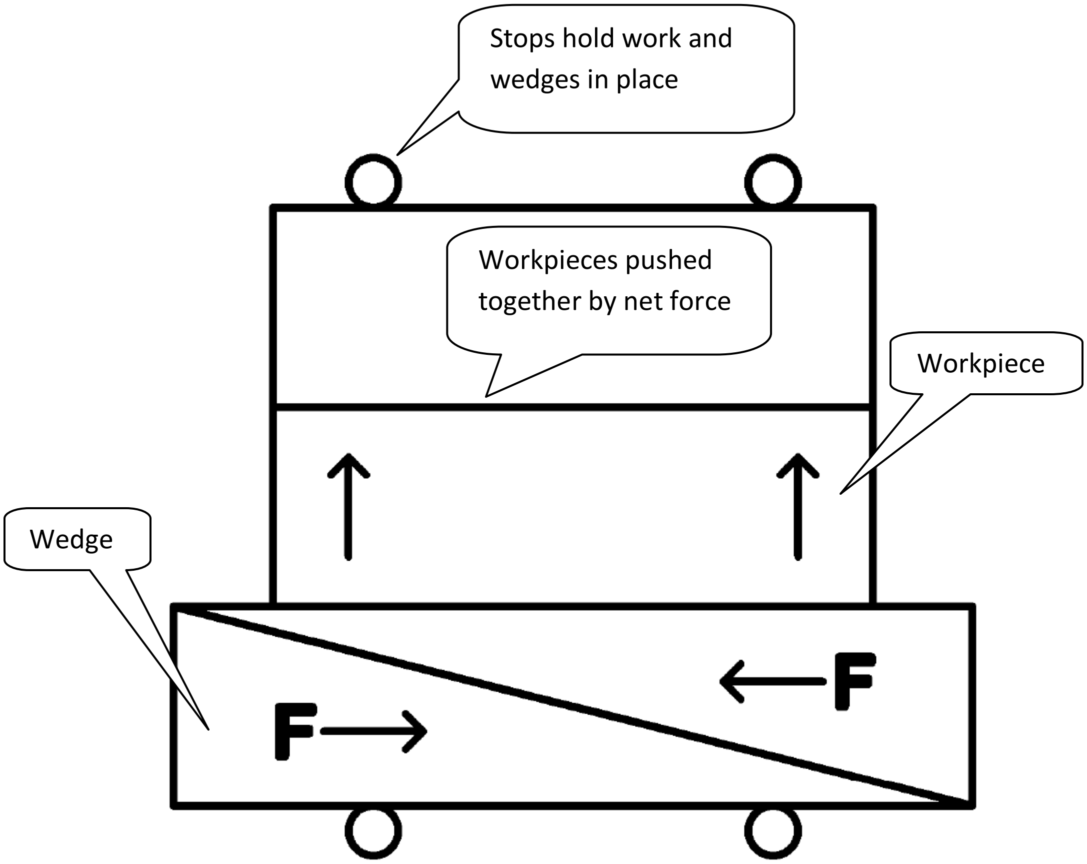

Figure 1 - Wedge clamps used to edge glue two boards.

Screw based clamping systems are the most common strong clamping methods and include vises, machine screws, and various types of hand clamps including C clamps and F clamps. Because a screw is designed for large mechanical advantage in a small space, and is easy to build rigidly into a moving system, screw based clamps are the preferred solution when high clamping forces and fixture rigidity are required. If lathe chucks are included in this category, then the vast majority of all clamping done in both machining and woodworking is done with screw based clamps.

The common types of screw based clamps found in a modern shop are vises, machine screws, and clamping sets. Clamping sets, shown in Figure 2, operate by placing one end of a clamp on a riser and pulling the other end down onto the work with a screw. The most common clamping sets use ½" screws, but they come in many different sizes. Vises are an excellent choice for high holding forces, capable of providing over 11,000 pounds of clamping force on objects 6-8 inches across, with 6000 pounds of force and

1500 psi being normal values for the most commonly used machine vises (MillVises.com, 2011). Vises only apply force in one direction and can have issues with deforming workpieces during clamping, which can lead to loss of accuracy. Vises also have to be relatively large compared to the size of workpiece they are capable of clamping, thus tending to be less space efficient than other options. Screwing a workpiece down directly as a method of fixturing works very well, but it requires that the workpiece have holes through which screws can be inserted. Clamping sets allow screw clamping to be used even on parts with no through holes. Screws and clamping sets are both quite versatile in terms of workpiece size and shape, but require external features to index the workpiece in place as they provide no guarantee of repeatable location themselves. Vises are often used with 'soft jaws', which are screw on jaws with indexing features machined in to them. Each part is inserted in the same place, allowing the process to be executed identically over and over without needing to verify workpiece location each time.

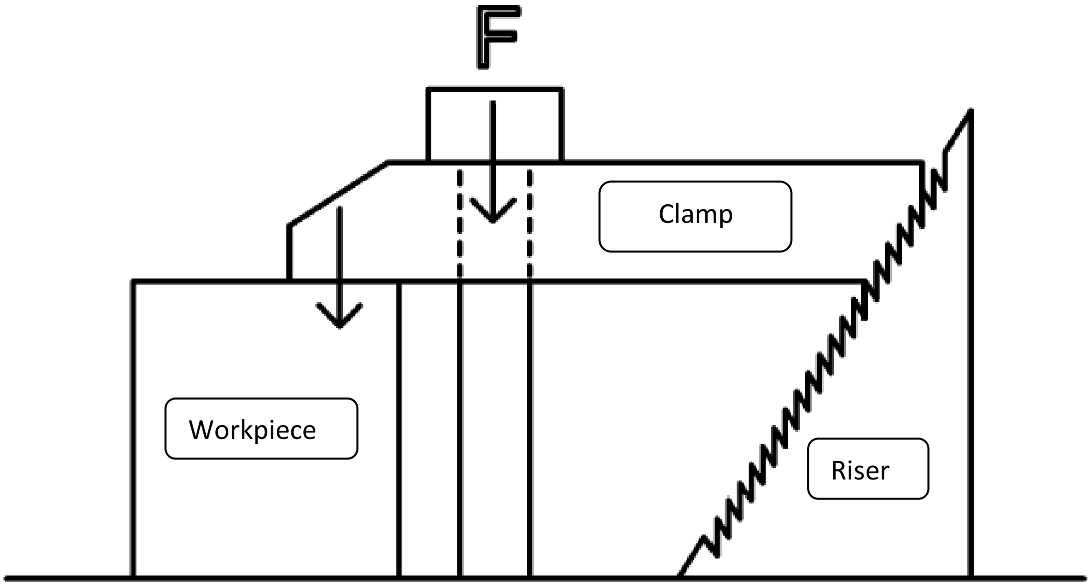

Figure 2 - Clamping set operating principle

Springs and elastic bands are a common method of clamping mechanically, and are one of the quickest and most affordable ways to hold a workpiece. On the other hand, generally clamping systems based on springs and elastic substances have low mechanical advantage and so the operator must be capable of generating a significant part of the total clamping force to apply or remove the clamps. This is in sharp contrast to wedge or screw based systems where the force input into the clamp is multiplied as it is applied to the workpiece.

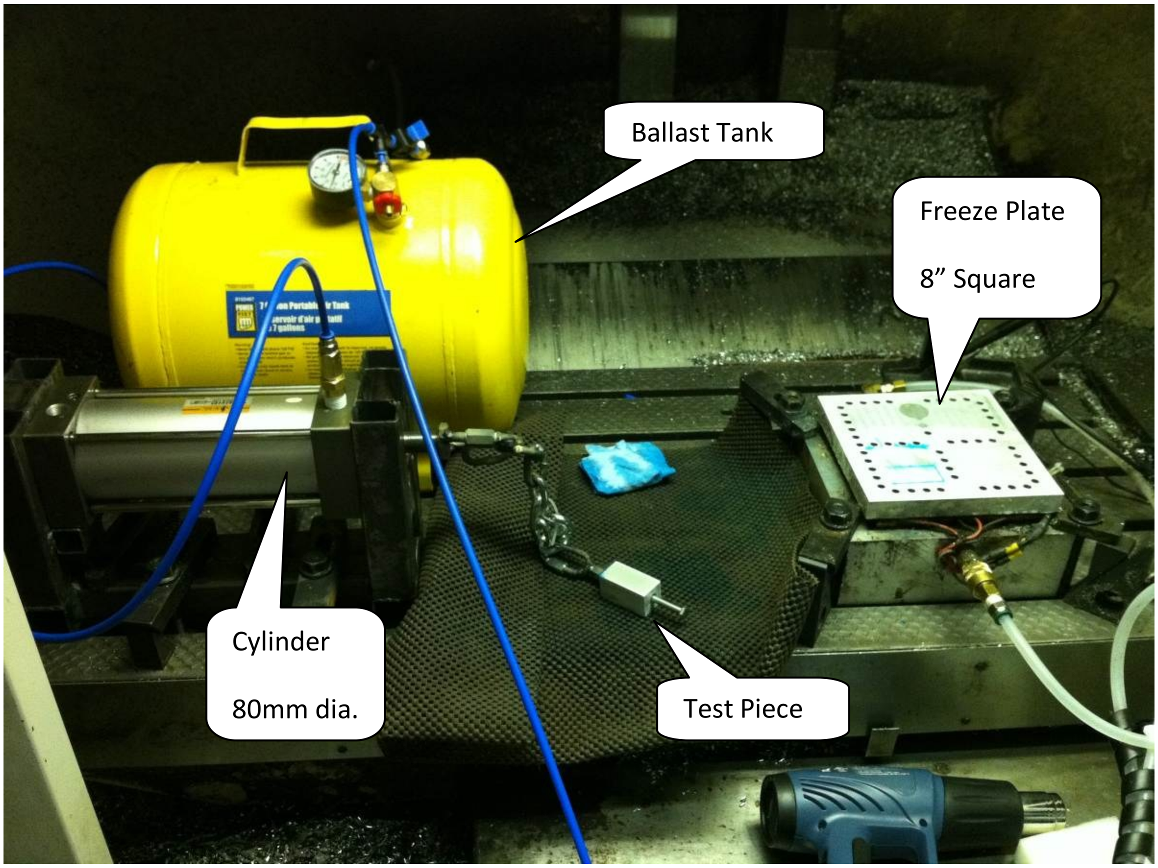

Figure 3 - Compound image and foreshadowing: pneumatic cylinder is on the left and a clamping set (black) is used to hold down the freeze plate at right.

Air cylinders and hydraulics can be used in place of screws in vises and clamping sets, and have the advantage of being able to actuate quickly or provide even more clamping force than possible with screws. The disadvantage is that they tend to be larger than their purely mechanical counterparts, and require pumps and hoses to be run to the moving components. An air cylinder and clamping set are shown in Figure 3.

Mechanical clamping is the first choice when available and is appropriate in one incarnation or another for almost all metal and plastic parts being manufactured. The exceptions to this rule are parts which are thin and flat and parts which don't have the structure to be squeezed from any angle without substantial deformation. Clamping force in vises and other types of mechanical clamping are often poorly controlled and can often lead to difficult to diagnose variance in part dimensions which can't be accounted for otherwise.

2.2 Adhesive Clamping:

Adhesives have some distinct advantages over mechanical clamps when it comes to working with complex shapes and work which is delicate. Adhesives generally require very little force to attach a part, but hold firmly once cured, which allows parts which would not stand up to the crushing forces of mechanical clamping to be held. Adhesives can hold onto a very large surface area of a part, essentially whatever can be brought into close contact with the surface being fixtured to, which allows the average holding force per area to be low while still providing a large net holding force. Adhesives also work well for work which is too small to carefully apply mechanical clamps to, as fixturing the work can be as simple as placing the work in a puddle of adhesive. Adhesives have the advantage of being the only type of clamping which can allow machining all the way around a part with no holes while having strong shear strength.

The disadvantage of many adhesives is that releasing their bonds can be difficult, since a workpiece is no more capable of enduring stress after being machined than before. Therefore, the most effective adhesives for fixturing use are those which release readily with heat or solvents. Pressure sensitive adhesives are also commonly used as they can often be released by careful peeling, which lets the user take advantage of the net holding force while removing parts with a small per area force. Peeling requires flexible workpieces and careful planning and is a much less predictable procedure than other methods of releasing work.

Hot melt adhesives are used in jewelry and woodworking production, and specialized systems for machining. Their main benefits are the ease of application, and reasonable adhesion to most substrates, while leaving no residue on release. The low peel strength of hot melts allows them to work well in situations where the joint is put in shear during work, but then released by putting the joint in tension.

Some adhesives are shock sensitive, meaning they have high shear strength in normal conditions but fail very cleanly under shock. Cyanoacrylate (CA or ‘superglue’) and hide glue both have this property, making them preferable for some types of work with odd and delicate parts. CA is readily dissolved by acetone and nitromethane, and hide glue is easily released by moisture and heat. Hide glue is used by guitar makers and violin builders on joints which need to be strong but might need to be disassembled later, and as an added benefit these joints will come apart cleanly if an instrument is dropped, allowing an easy repair. Both hide glue and CA can handle the stresses of machining a component in wood, shell, and even metal but can be released by quick shock load produced with a hammer and a block of softer material to prevent marring.

Adhesives are ideal for exceptionally flat parts with no thick edges for mechanical clamps to hold on to, and the entire top face of a part can be worked on without worry of a clamp collision. Double sided pressure sensitive adhesive (PSA) tapes are known in machining circles as a ‘secret weapon’ for components like these, though there are caveats as most strong PSA tapes leave adhesive on both surfaces when separated and the adhesives are harder to clean than most liquid adhesives. For parts such as aluminum honeycomb and other filigree type parts, and small brittle components made from such materials as ceramic and graphite, adhesives are essentially the only choice available. Chapter 3 contains a more thorough overview of adhesives in terms of types and usage, theoretical background, and history.

2.3 Physical Process Clamping:

This category includes a number of novel clamping methods that don't fit well in other areas. These include clamping based on pressure differentials, thermal expansion, and magnetic clamping.

Thermal expansion and contraction are reversible processes capable of exerting tremendous force. Bearings and pulleys are fitted to shafts through use of these principles, often heating up one part and freezing the other and allowing interference based on returning to their previous dimensions to hold them together. This principle can be used for fixturing components in exactly the same way where they will be released by heating or cooling the assembly. Another use of this effect in machining is in 'heat shrink' tool holders, which are heated up to expand their bore and then contract around a tool shank as they cool.

Expansion by drying and wetting is also an effective method of causing an interference fit in a joint. The oldest use of this was drying out wooden plugs, used analogously to nails, and placing them in holes through two components which were just large enough to accept the dried plugs. As the plugs reacclimated to normal humidity they would expand and provide an interference fit. The same effect was used to split stone in quarries as holes were drilled in the stone, filled with wooden plugs, and then water applied to the plugs to cause expansion and split the stone. This process is rarely used in manufacturing, but could be effective in the right application as it provides extreme force and has no release requirements beyond a dry room.

Figure 4 - Braces are glued to a guitar top inside a vacuum bag, (Scott, 2011)

Clamping by air differential is more common in industry. Holding via air bladders expanded against a part is commonly used in glue-ups in woodworking industries. Vacuum clamping is even more common in both woodworking and metalworking shops. In vacuum clamping, a part is placed on a fixture with a gasket seal around the contact area and air is evacuated from beneath the part, or alternately two parts to be joined are placed in a flexible bag which is evacuated as in Figure 4. Atmospheric pressure is thus removed from one side of the part providing a hold down force equal to 1 ATM, or roughly 14 psi, at sea level. Vacuum pumps are inexpensive, as low as $60 for air powered units and $200 for mechanical pumps, and the clamping forces can be immense for workpieces with large surface area. In commercial woodworking, cabinetmaking, large panel, and aerospace manufacturing, vacuum workholding is standard operating procedure for most large parts. The disadvantages of vacuum workholding include the requirement of a flat holding surface or a fitted fixture, limited hold down force on small parts, low shear resistance, and extremely inelegant failure vectors- vacuum tends to fail all at once, at which point workpieces often take flight. The author has observed many a workpiece take to the air as vacuum has been lost due to leaks or material failure. However, vacuum workholding is far faster in turnaround than any other method, taking less than a second to clamp or release a component. It is also extremely space efficient as parts can be nested together with just enough distance between them to navigate the tools used in the process.

Clamping with powerful electromagnets is popular in the ferrous metal processing industry. Magnetic chucks are weaker than mechanical fixturing, but powerful enough for moderate material removal rates in magnetic steels and iron. Magnetic fixturing is extremely common in grinding applications where the workpiece geometry tends to be flat and the cutting forces predictable. Magnetic holding forces are roughly fifteen times higher than vacuum holding forces, but their use is limited to ferrous metal and the holding forces depend on the quantity of iron in the metal (Demeter, 2004).

For large sheet processing and grinding, the physical clamping methods reign supreme. Vacuum clamping is also very popular in industries with smaller machining forces for working on 3D parts. Nearly all guitar parts which are manufactured using automated machinery are held using vacuum fixtures, and vacuum has substantial penetration into other woodworking industries such as turning and ornamental architecture.

2.4 Compound Fixturing:

Compound fixturing is attachment of a workpiece to another object which is more easily fixtured. Examples of compound fixturing include potting, onion skinning, and tabbing.

Potting is the process of casting the workpiece in a block of some removable material. Common potting materials include wax and low melting point alloys, commonly known as ‘cerro’ or fusible alloys for their ability to be easily melted and reconstituted (Bolton Metal Products, 2010). Cerro alloys contain bismuth as a primary component and can have melting points as low as 47 degrees Celsius. Cerro is used to fill pipes before bending to prevent kinking and collapse of the inner surface, and then melted out. Cerro is used in fire safety equipment as seals on sprinklers, and as an adhesive in lens manufacture. Generally, parts are potted completely in a solid block of cerro and the cerro is melted off after machining. As cerro is quite dense, the chips produced during machining can usually be heated and the cerro recovered by skimming the other metal chips off. This process is used in the manufacture of impellers and other fragile parts in the aerospace industry. The main disadvantage of potting is the speed of the process, and the effort required in recycling the material. This large time consumption makes it impractical in most situations.

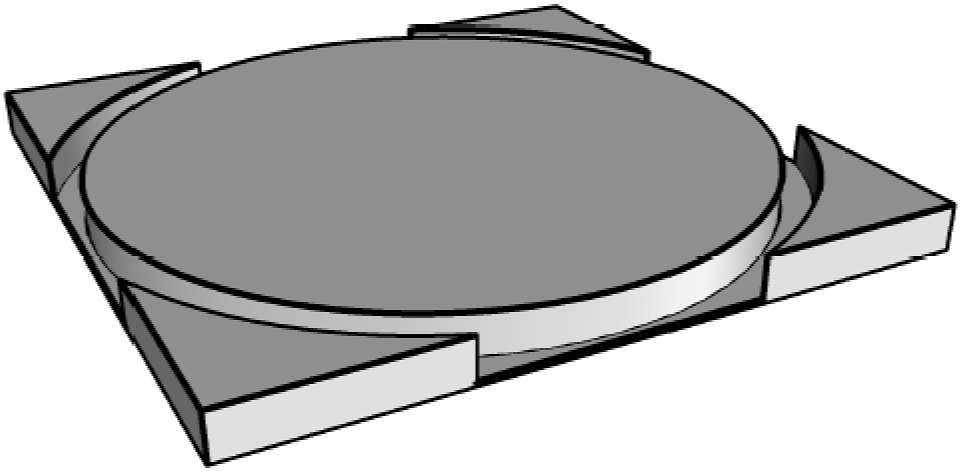

Figure 5 - Onion skinning of circular workpiece. Skin is left around piece, and clamps can be used to hold onto the four corners of the stock.

Onion skinning, shown in Figure 5, is the practice of leaving a component attached by tabs or a thin skin of material to the rest of the stock- the remaining stock can then be held by mechanical clamps. Onion skinning is quite common in woodworking and production of certain small metal components, but it greatly increases finishing time as the tabs must be removed by cutting or sanding them off. Onion skinning uses stock extremely efficiently as the only space needed to be left between parts is the width of the cutter used for their outside profiles. Tabbing is another form of onion skinning where the piece is mostly cut through, but tabs of material are left between workpieces and the bulk of the material. Tabbing is used in place of onion skinning in processes where there is no control of depth of cut, such as laser or water jet cutting. A typical tabbed workpiece is shown in Figure 6.

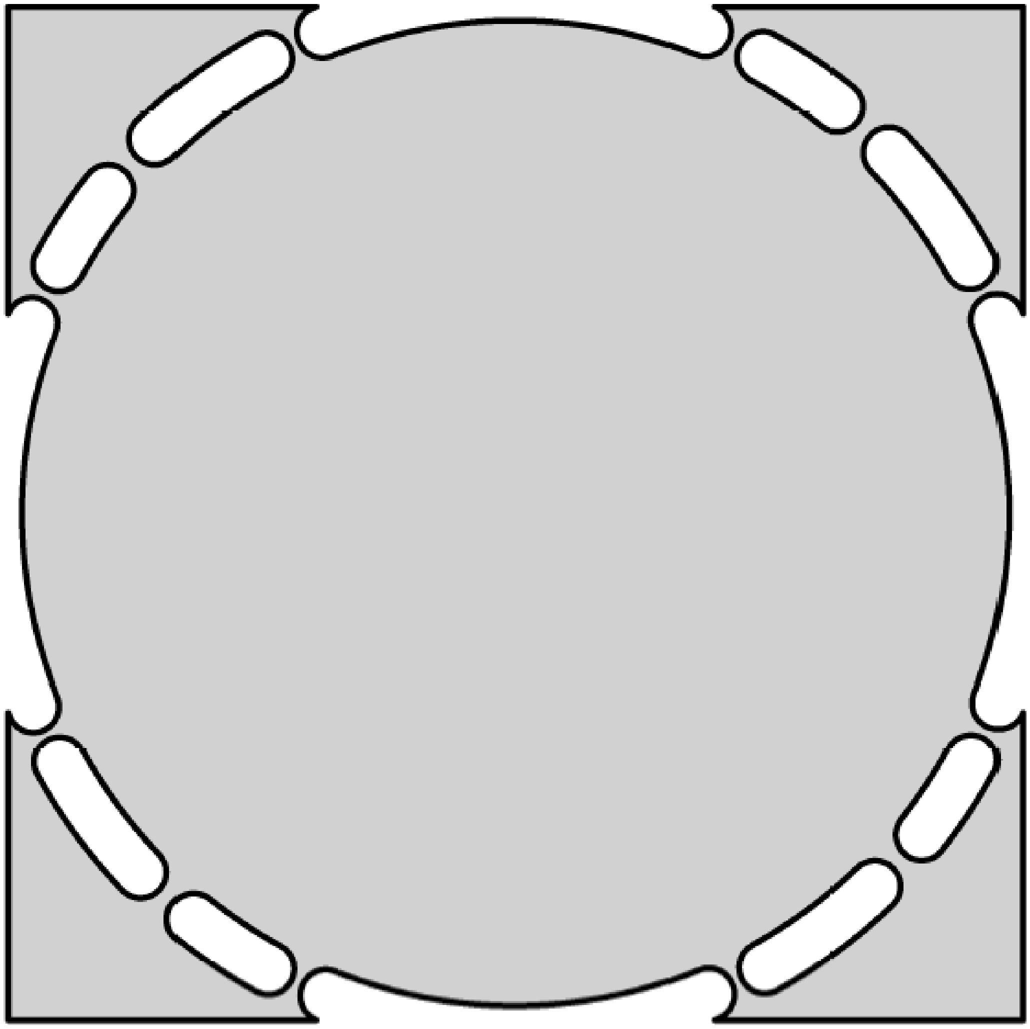

Figure 6 - Workpiece similar to Figure 5, using tabs instead of onion skinning.

2.5 Ice as a Fixturing Medium:

Ice fits into multiple categories as a fixturing medium. It has been used successfully as an adhesive, but is also an excellent support material and therefore can be used for potting workpieces. Ice can work in mixed mode adhesive/support roles as shown in Figure 7, where the ice adheres the part to the fixture and works as a support material around the part to further resist shear forces. The temperature of ice can be of benefit both by changing material properties to make soft materials stiffer and more machinable (Shih, Lewis, & Stronkowski, 2000), and keeping work and tool temperatures down by cooling the entire system. This is noticeable when machining easily melted materials such as plastic and aluminum. On the downside, if high accuracy is required then the thermal expansion of the material must be accounted for in the production process design.

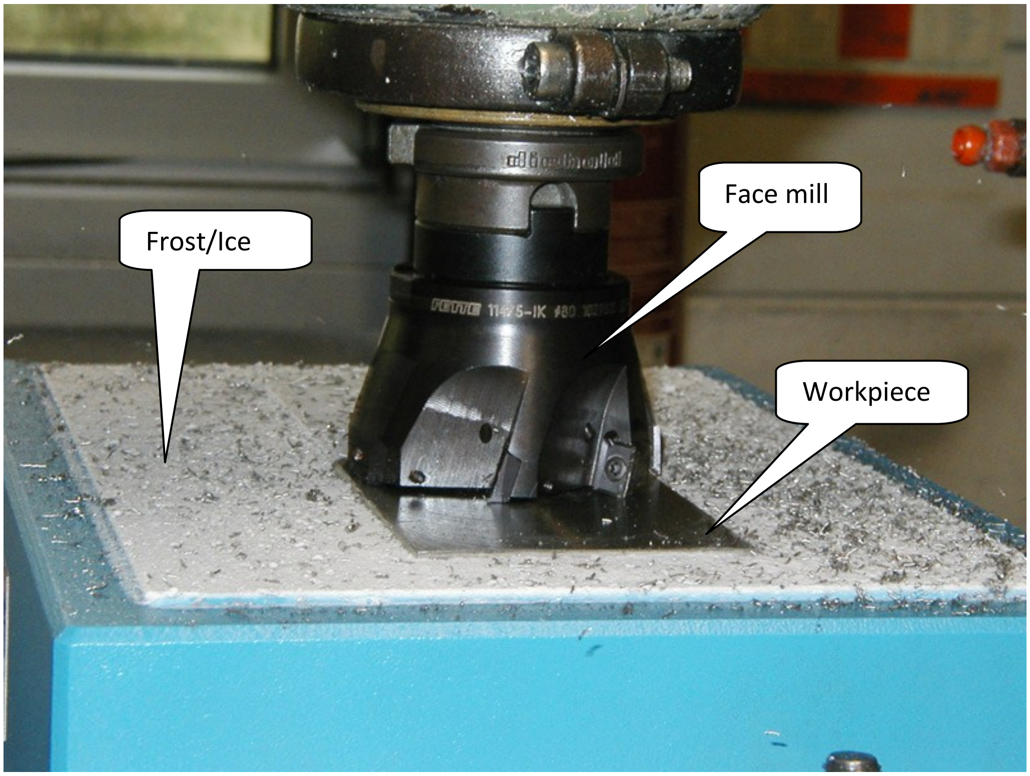

Figure 7 - Ice fixture holding part being face milled. The ice layer is almost invisibly thin, but frost covers the cooled area (Horst Witte, 2010)

Ice is slower than other adhesives used in fixturing. Ice takes one to two minutes to freeze or thaw, versus tapes and CA which can be laid down and released almost instantly (Demeter, 2004). On the other hand, ice melts away cleanly and doesn't leave any residue on the parts or the fixture. In the case of double stick tapes and CA, cleaning left over residue can be a difficult job and if the residue is not cleaned then part positioning can be affected. Ice is also extremely cost effective and environmentally friendly. Even the gentlest adhesives require physical force, strong heat, or long immersion in solvents to release. Conversely, the release force of ice on the part is effectively zero and requires nothing more than returning the system to room temperature.

Ice requires more expensive equipment to work than any other adhesive, given the temperature change that must be effected, regardless of the particular system used. Systems using conventional refrigeration require separate refrigeration units weighing over 60 kg(Horst Witte, 2010), air powered systems require substantial amounts of air and thus a large compressor, and thermoelectric systems require both electricity and a means of cooling themselves.

In the end, ice becomes economical as a replacement for adhesives on components and materials which are too fragile to be held mechanically, and for which potting processes are too slow or too expensive to set up. Micromachining of small ceramic and graphite parts, and aluminum honeycomb, are mentioned as common uses by the manufacturer. In the author's own experience as a manufacturer of extremely fragile shapes in shell (nacre, wood, and metal for the musical instrument inlay industry, ice can be a viable alternative to other adhesives and offers numerous advantages.

2.6 Fixturing Wrap Up

Just by merit of their continued used, it is clear that different fixturing methods all have advantages in specific niches. For workpieces with square edges, the vise is the clear winner whereas for very large sheet workpieces vacuum has more to offer than any other fixturing method. Table 1 shows a general comparison of common fixturing methods. Bases for comparison include the ultimate holding force, the force applied to the work during application and removal of the fixture, the speed of the fixturing method, and the cleanup required after use. Application and release forces, the forces placed upon the part when placing in and removing from the fixture, are often the determining factor in whether a fragile workpiece can be held with a particular method.

Table 1 - Comparison of common fixturing methods

Clamping Method	Holding strength	Application force	Release Force	Speed	Cleanup
Vise	Very high	Very high	-	Fast	None
Toe Clamps / Screws	High	High	-	Very slow	None
Vacuum	Low	Low	-	Instant	None
Magnetism	Medium	Low	-	Instant	None
Adhesives	High	-	High	Very slow	Substantial
Onion Skinning	Medium	-	Low	-	Substantial
Ice	Medium	-	-	Very slow	None

Chapter 3 – A Stereotypically Sticky Subject: All About Adhesives

As the topic of this research is use of ice as an adhesive, it would seem imperative that a thorough overview be given to the field of adhesives in general. An overview of different adhesive types is presented, followed by a technical review of the scientific basis for adhesion and how this can be used to optimize adhesive joints.

Besides understanding the adhesive and material being used, surface preparation is paramount in achieving good joints and different techniques will be covered in some detail. The fundamental processes governing adhesion, wetting and surface energy, will be covered in some depth.

3.1 A Brief History of Glue:

Man's use of adhesives predates history: tar from birch tree resin was used 200,000 years ago to make spears (Wadley, Hodgskiss, & Grant, 2009), and 70,000 years ago compound adhesives were being made from plant gum and red ochre. In fact, these adhesives are used as evidence that humans were having complex thoughts far earlier than they could surmise purely from examining use of symbols in art or cave paintings. In fact, humans were engineering adhesives for 65,000 years before they figured out how to smelt metal (Holmes, 2008).

About 6000 years ago, humans began using animal based glues made by rendering animal products to retrieve sticky collagen proteins contained in them. Many objects constructed with these animal glues have survived today, and the glue itself, now known as hide glue, is still a favourite of musical instrument builders and some woodworkers. Hide glue is so named because it is made from animal hides, hooves, and connective tissue which is reduced to jelly and then dried and distributed as flakes or granules. Hide glue was in fact the only glue available until World War 1, and so all antique furniture and woodwork and early aircraft were constructed using it. Besides its hardness and quick setting, hide glue also has the characteristic of being notoriously vulnerable to failure when exposed to moisture.

During World War 1 glues based on casein and nitrocellulose were first developed, and advances in chemistry during the 1930s lead to a large number of new synthetic resin glues. During World War 2 a number of advanced synthetic adhesives were developed including cyanoacrylates, commonly known as super glue, and epoxies. These were strictly for military use initially, but became available to wider industry and consumers during the 1950s and 60s.

3.2 The Basics:

Adhesion is a process of attraction that causes dissimilar materials to cling together, while cohesion is the same process between similar molecules. At a basic level, all adhesion is the result of molecules in intimate contact with one another and held together by various forces. By far the most important of these forces is known as dispersive adhesion, or physisorption. This is the attraction caused by the intermolecular or Van der Waals forces, the attraction between positively and negatively charged poles of molecules in different substances.

An adhesive is a mixture in a liquid or semi-liquid state which can adhere to and bond surfaces together. Adhesives can cure or harden by evaporating solvents, temperature change, or by way of a chemical reaction between the adhesive components or the adhesive and a catalyst. All adhesives function by polymerization, which is the process through which molecules in a substance bind together into long chains, making the substance harder. The adherand is the surface of substance the adhesive is bonding to.

3.3 Common Adhesives:

Adhesives can be broadly categorized into two classes: non-reactive adhesives which cure without a chemical reaction, and reactive adhesives which require a chemical reaction to cure (Todd, Allen, & Alting, 1994). Examples of the first category include wood glue and hide glue, which cure by evaporation of their water content, and hot melt glue which cures by cooling. Examples of reactive adhesives include two part glues such as epoxies which will remain uncured indefinitely unless combined, and UV curing adhesives which will only cure in the presence of a strong UV light.

The most common non-reactive adhesives are drying adhesives, which cure by evaporation of a solvent contained in the adhesive. Common drying adhesives include white glue, wood glue, hide glue, and contact adhesives. While the first three form bonds simply by polymerizing as the solvent evaporates, contact adhesives have a more interesting method of forming a final bond. Contact adhesives are applied to both surfaces to be joined, and allowed to cure by evaporation. When the surfaces are brought together the two layers of contact adhesive undergo a process called strain crystallization where the strain on the adhesive causes much of it to change phase from an amorphous (flexible) solid to a solid crystalline form. Natural and neoprene rubber can both function as strain crystallizing adhesives.

Pressure sensitive adhesives (PSAs), the most common form of which are adhesive tapes and stickers, form a bond by being soft enough to form closely to the adherand when pressure is applied. The term pressure sensitive is not a misnomer- the pressure applied to the adhesive over its surface contact area is directly related to the bond strength (up to the materials adhesive limit). PSAs can form strong bonds as they are also hard enough to resist flowing when stress is applied across the bond. When the adhesive and the adherand are in close enough proximity, the intermolecular forces can cause the bond to become much stronger, and some PSAs are designed to be permanent after an initial setting time.

Hot melt adhesives, also known as hot glue or thermoplastic adhesives, are thermoplastic compounds applied in liquid form which solidify upon cooling to form strong bonds on a wide range of materials. Hot melt glues have numerous advantages in ease of use and application, being water resistant, unlike many other types of glue used in soft-material manufacturing including foot wear, apparel, and packaging.

Solvents can be effective adhesives for certain plastics. Acetone is excellent ‘glue’ for acrylic and results in clear joints. Many plastic cements are actually just samples of the plastic dissolved in the proper solvent. Acetone dissolves many common plastics and is safe, so it is a good place to start, though many plastics will require nastier solvents. Most general plastic cements are just thickened versions of a mixture of strong solvents. Properly executed, a solvent joint is more of a ‘weld’ than an adhesive joint as the two sides of the joint mix and form what is called a diffusive adhesive joint. This is the strongest possible joint between two materials as they actually mix into one another.

The most common reactive adhesives by far are epoxies and polyester resins, along with polyurethanes. Epoxies come in an enormous variety of formulations with viscosities from water thin up to putties, cure times from minutes to days, and a range of cured hardness from flexible to crystalline. Polyurethanes come in formulations from the common construction and foaming adhesives which are catalyzed to cure in the presence of moisture to two-part casting resins that exhibit a wide range of hardness and flexibility. Because polyurethane is a good adhesive and a casting resin, it has been used to cast in place the protective leading edges on wooden airplane propellers. Due to their highly controllable and predictable cure times, epoxies and polyesters are the mainstay of the composites industry with the terms fiberglass and carbon fiber usually referring to glass strands in polyester resin and carbon fibers in epoxy respectively. UV sensitive catalysts, which react to the presence of a strong UV light, have been developed for polyester and epoxy resins and others and have become important in medical and dental procedures and optics and electronics manufacturing.

Another important reactive adhesive, and a personal favourite of the author, is cyanoacrylate also known as superglue or CA. CA glue comes in a number of viscosities, and even flexible formulations, made by adding soluble rubbers or fillers to the CA resin.

CA is a mainstay of small parts assembly due to its almost instantaneous cure, and the major component in 'liquid stitches' and 'liquid bandage' products. Though relatively benign, CA is believed by many to be highly toxic due to the extreme burning caused by the fumes entering the eyes or mucous membranes in the nostrils. The sensation is actually caused by the airborne CA curing on the eyes or membranes. Another popular non-adhesive use of CA is in fuming for fingerprints or other organic residue- police or researchers put the article in question in a chamber with heated CA resin and the fumes cure on any oils or moist surfaces present leaving a whitish haze.

3.4 Making Good Adhesive Joints:

Though all of the terminology in this section will be explained in sections 3.6 and 3.7, the majority of the factors which contribute to good adhesive joints may be accounted for without a strong understanding of the technical details. It is for this reason that the practical aspects are explained with an emphasis on applicable clarity rather than technical rigor.

There are a lot of misconceptions about how adhesion actually works, and a little understanding can go a long way in achieving superior results. The first step is to understand what is going on when one is attempting to make a good adhesive joint, so a basic model is presented here.

The best way to make a good joint is to avoid making a bad joint. This means knowing the ways joints fail and how to avoid them. Luckily for us, there are only two ways in which a joint can fail due to improper adhesive use- adhesive failure and cohesive failure. Adhesive failure, when the adhesive breaks free of the substrate, is caused by unclean joints and poor wetting. Cohesive failure, when the adhesive itself shears leaving adhesive on both substrates, happens when a joint is improperly fitted leaving excess adhesive in the joint or, even worse, not enough!

3.4.1 Wetting:

To work well, an adhesive needs to 'wet' the surface it is bonding to (Kendall & Kendall, 1994). An easy way to see wetting in action is to notice that water beads up on some surfaces and seems to form sheets on others. The water that beads up does so because the surface tension of the water, the attraction of the water molecules to each other, is stronger than the attraction of the water molecules to the surface. In the case of water sheeting on a surface, the water is more attracted to the surface and so it 'wets' the surface and spreads out into a sheet. To form a good bond, the adhesive must be attracted to the surface, and that means good wetting.

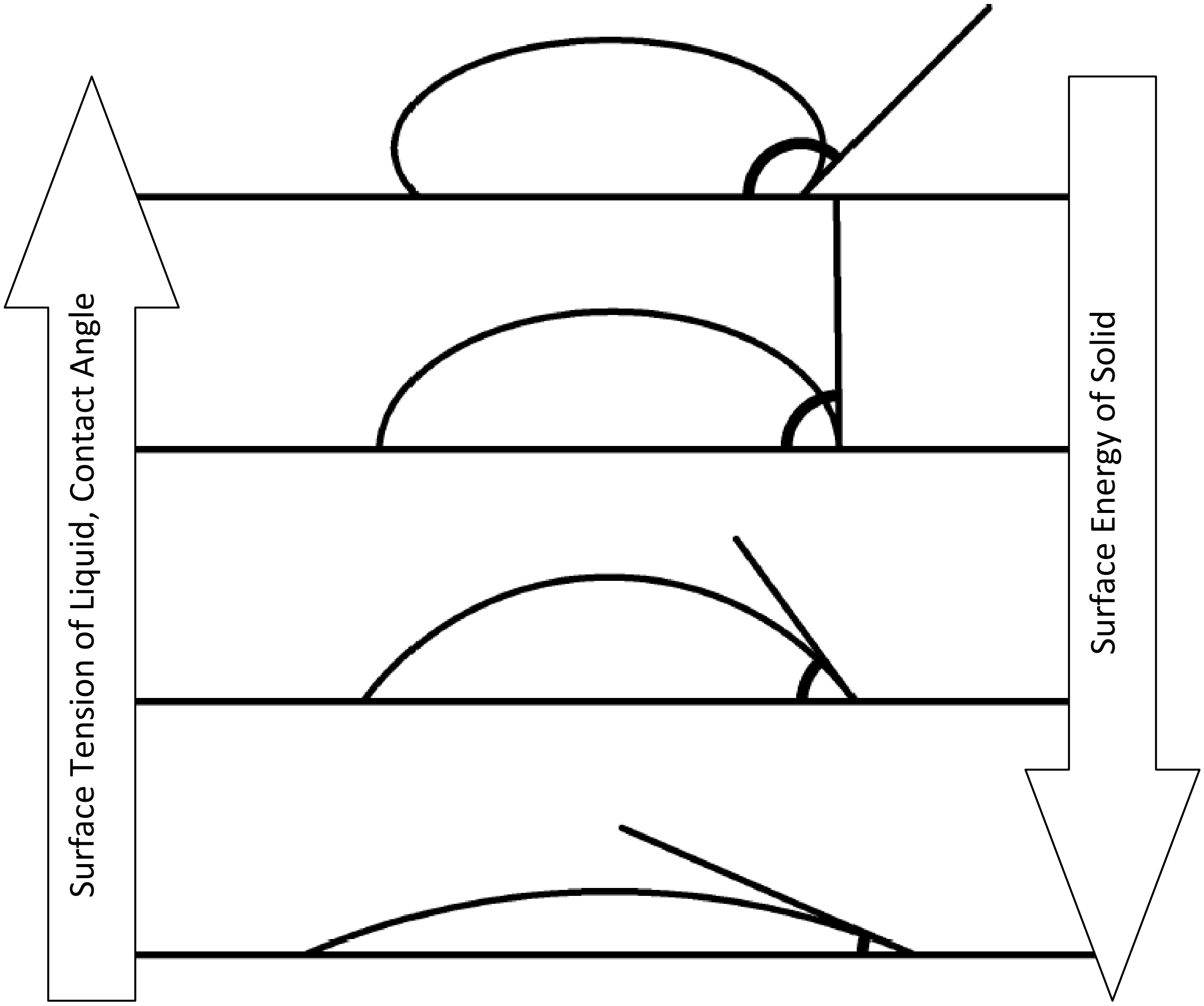

Figure 8 - Example of a drop wetting a surface. Contact angle with surface is shown to right of drop.

So, if wetting is so important then how can one tell if a surface is ready to be bonded? It turns out that the standard test is to spray distilled water on a surface and see if it beads up or flattens out (SmartAdhesives Inc., 2011). An example of results from the spray test is shown in Figure 8. Drops will flatten out on a surface with decreasing surface tension of the liquid or increasing surface energy of the solid. There can be false positives if a surface is not clean, but on a clean surface the water test is quite reliable. Often, even with surfaces most people would assume are always ready to glue, the water test will come up negative. A common example of this is wood, but even well-fitted joints on wood surfaces can be surprisingly weak without proper surface preparation.

Getting good wetting is a combination of making the surface 'active' by increasing surface energy, and removing contaminants that get between the adhesive and the surface. Surface energy can be thought of as the attractiveness of a surface to other substances- the more energy is on the surface, the more it can pull in the adhesive and react with it. Surface energy can pull in not only adhesives but other substances such as oils and contaminants, or even the oxygen from the air, and so if a surface sits for long enough its surface energy will diminish as the surface finds things to react with. On substances like wood it can take hours or days for the surface energy to disappear, whereas on some metals like aluminum the reaction with the air is so fast that the surface loses all its energy in minutes! Low surface energy leaves nothing for the adhesive to grab on to, so it just ends up sitting on the surface rather than sticking to it.

To make a surface ready for bonding, it is best to work from the top down through the layers of contaminants that get between the adhesive and the fresh, reactive part of the surface. From top to bottom, these layers are the loose layer, oils and grease, films and oxides, and the reactive layer of the substrate. A diagram of the layers is shown in Figure 9.

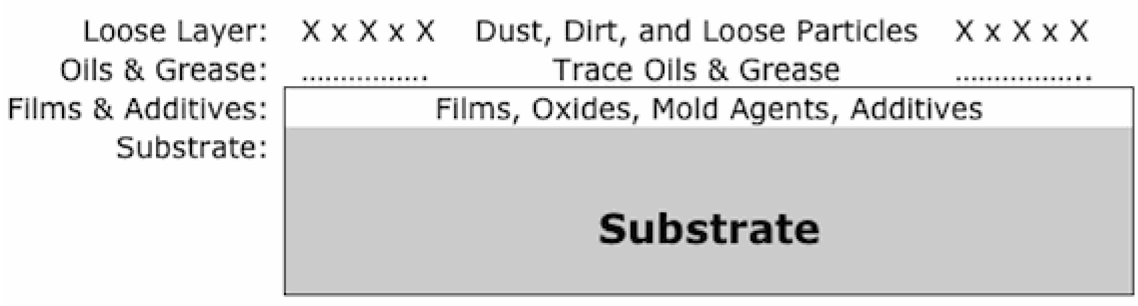

Figure 9 - Hierarchy of surface contaminants, (SmartAdhesives Inc., 2011)

The loose layer contains dust, dirt, water, and loose particles of the material and can cause a false positive on the water test as the water flattens out by reacting with the waste material. Even on 'clean' material, the loose layer can often be composed of fine metal or wood dust from the substrate itself and so it is important to wipe down or blow off the surface with compressed air. In general, everything in the loose layer can be wiped off or will evaporate on its own.

The oils and grease layer contains substances which will not evaporate on their own and cannot simply be wiped off of the surface. Oils tend to have very low surface energy, and so they cling to surfaces tenaciously even in the thinnest layers. In the case on nonporous surfaces, such as metals, one or more wipes or a bath in a volatile solvent is the best way to ensure a clean surface. Acetone and isopropyl alcohol are safe solvents and are preferred in most cases, isopropyl alcohol mainly for plastics which are soluble in acetone. In some cases where there is serious contamination or neither of the previous solvents will work, methyl ethyl ketone (MEK) and other dangerous solvents may become necessary. In the case of porous surfaces and spotty oil contamination, as is seen in some woods, a solvent wipe can turn a partially contaminated surface into a fully contaminated surface. Experiments have shown that it is effectively impossible to remove contaminants contained throughout the porous substance with surface wiping. The suggestion in these cases is to cleanly cleave the surface by planing or careful abrasion meant to remove surface material without smearing it across the new surface. As most woods internally are oily over only part of their area, this provides the maximum usable adhesion surface. User experience suggests that water soluble glues are more susceptible to failure due to oil contamination than other glues. Methacrylate based adhesives have a surprisingly high tolerance to grease and oil and are often marketed as requiring no degreasing on substrates (though it is still recommended).

The films and oxides layer contains hard films and the oxidized layer of the material itself. Hard films would include things like a galvanized or anodized surface on metals, or plastic coating and paint on various substances. As mentioned above, even non-metallic materials react with oxygen in the air to lower their surface energy, and so everything has an oxidized layer which must be removed. The normal method of removing the films and oxides layer is abrasion with fine sandpaper in the 120 to 220 grit range or steel wool / bonded abrasive or sandblasting. It is important to remove all loose particles after abrasion with compressed air or vacuum as these constitute a new loose layer. In materials where there is a substantial films and oxide layer, such as highly oxidized metal, it can be necessary to abrade the surface first and then go through all the steps above a second time as the oxide layer might 'hide' contaminants from the earlier steps. An alternative to abrasion is shearing off a layer of material by using a cutting tool such as a milling machine or planer. It is important than this be done without oils or coolants as those can contaminate the surface and reduce the surface energy.

In the case of certain substances with extremely low surface energy, it can be necessary to alter the surface of the material beyond exposing fresh material. Some materials such as polyethylene, acetal, nylon, aluminum, copper, and stainless steel are simply difficult to bond as they either react with oxygen in the air almost instantaneously or they are chemically inert to begin with. For these materials it can be necessary to alter the properties of the surface itself by using chemical primers, etching the surface with acid, scorching the surface, or grit blasting. Other treatments include plasma and ion guns, which are often used in industry as pretreatments for painting plastics and metals.

In the case of metal surfaces, the standard procedure is to degrease the surface to remove loose contamination, followed by abrasion, and then another round of degreasing. Many metals require an acid etch after the second degreasing and nearly all require the adhesive to be applied as quickly as possible after treatment.

Plastics should be treated as metals, though usually they only require abrasion followed by degreasing. Some plastics which can absorb contaminants, particularly composites using polyester or epoxy, need to be degreased twice. Plastics must be degreased with a solvent which does not attack the particular material in use, so care must be taken. Low surface energy plastics can require an acid etch or plasma treatment.

Wood and other porous organic materials are usually fine with abrasion followed by thorough cleaning off of dust. Planing is preferred as it leaves less dust on the surface, though blowing the surface off with strong compressed air makes the two effectively equivalent. There is some evidence that planing is less likely to spread internal oils across the wood surface.

Application of adhesive to all surfaces will ideally verify full wetting before a joint is assembled. Though it is standard and ‘good enough’ in many industries to lay down a wave or bead of adhesive on a joint and verify joint coverage by ‘squeeze-out’ around the closed joint, this can leave pockets of un-wetted material inside the joint. The preferred technique is to fully coat both surfaces with adhesive, ensuring full coverage, and then bring them together. This can be difficult with fast adhesives like hide glue and CA, but in those cases it is still preferable to fully coat one surface if possible.

3.4.2 Fitting:

There are only a few mistakes to make with fitting a joint, but they can be easy to miss. It is common knowledge that a better fitting joint will be a strong joint, though the material and adhesive in question can factor into how good is good enough when it comes to fitment. Highly crystalline glues such as hide glue and CA have almost zero tolerance for poorly fitted joints due to their brittle nature, whereas glues which are strong and cohesive on their own such as epoxies can perform well even when filling large gaps. Some adhesives which expand on curing, such as polyurethanes, are perceived as being ‘gap filling’, but in fact the expanded glue is very weak structurally and the filled appearance of the joint can lead to a false sense of success. Glues in general are stronger in adhesion than cohesion, and so an ideal bond is one where there is minimal glue line thickness over which cohesive failure can occur.

Effective and common tests for fitment include holding or clamping the parts together and holding the part up to a bright light while looking for light coming through the other side of the joint. The human eye is very sensitive to light and this method can expose cracks that are otherwise too small for us to perceive. This ‘candling’ method, named for classical woodworkers who would use a candle as a light source, is highly effective in cases where a joint is relatively straight in contour but of limited use on curved joins. In the case of a curved joint, chalk or machinists bluing can be used as contrast to see where the two surfaces rub by gently moving them against one another. Chalk is often used by wood workers to test the fit of a blind joint.

Clamping errors can lead to a number of unpredictable problems with joints. Insufficient clamping pressure is a common issue with inexperienced workers and can leave a thick glue line susceptible to cohesive failure, the after effects of which are shown in Figure 10. Applying clamps too slowly with fast curing glues like CA and hide glue can lead to joint failure, often from a joint that appeared to ‘squeeze out’ properly, due to adhesive in the joint having cured before the surfaces were joined. These failures appear identical to adhesion failures, which they technically are, but the mistake was made in assembly rather than surface preparation.

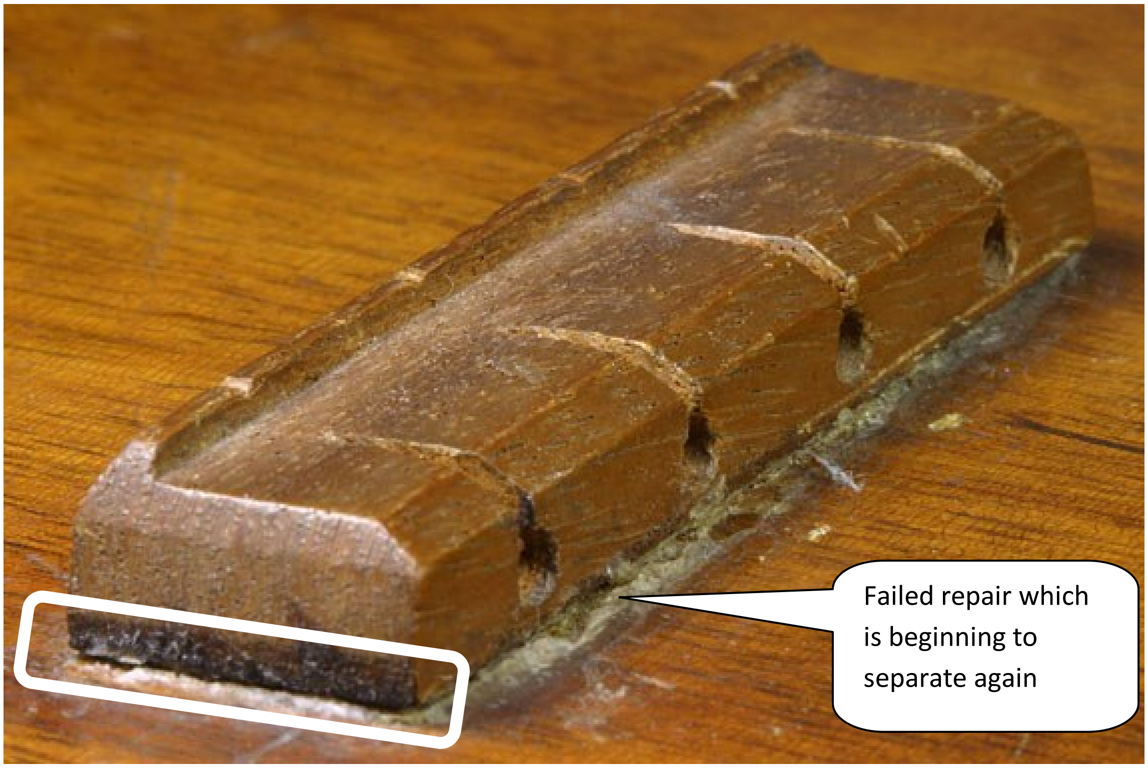

Figure 10 - A classic poor fitment failure, piece is glued onto a flat plate below. Gaps can be seen around the bottom of the piece being glued on (circled), and a repair was attempted by pushing more glue into the gap from the outside ((Ford, 2004)

A final kind of clamping error is uneven application of force. This one is often hard to spot even for experienced users. If clamps are applied and then loosened, a joint opens back up while having already squeezed out too much adhesive to fill the void. This most often occurs in glue-ups where a large number of clamps are employed; later clamps can actually loosen clamps already placed or force open the joint in an area where it had previously been closed.

3.5 Take Away:

When it comes to adhesion, cleanliness truly is next to godliness. There are a few simple steps to making a great adhesive joint, and yet the subtleties and misconceptions related to this simple task have items falling apart all over the world every day. The right adhesive and surface preparation will allow almost any two substances to be bonded together. The steps are easy, though implementing them can be a surprising challenge:

- Clean the surfaces of all dust, oil, grease, paint, coatings, etc. No one has ever regretted having too clean a surface for a glue joint.
- Activate the surfaces. For most materials this simply means exposing a new surface to the air right before gluing, but on some materials this can require acid etches, torch treatments, and even exotic treatments like corona or plasma etching.
- Verify your surface…water is cheap, broken joints are expensive, so do a wetting test. Verify your process to make sure the clamps all go on and that it can be done fast enough for the adhesive being used.
- Pick the right adhesive for the job. If time is limited, a fast setting adhesive is inviting disaster. Water based glues will swell and distort wood where other glues will not. Some glues will melt some substrates, and not in that good way where they melt together.
- Make good clean joints, and clamp them like there's a prize for how much glue you can squeeze out. The author and others have failed to squeeze enough glue out of a well wetted joint even with hydraulic pressures to make it fail, and the benefits of a tight joint are many.

3.6 Surface Energy Revisited

Surface energy is defined as the amount of energy required for a substance to form a new unit area of surface.(Athavale, 2010) In a liquid, surface energy and surface tension are identical in units and value, and this makes understanding the concept of surface energy easier. The surface tension can be thought of as the force attraction of the molecules of a liquid to each other. Water has a surface energy of 0.072 J/m^2 and a surface tension of 0.072 N/m.

A sphere is the smallest surface area configuration for given volume of material, and is the shape that a liquid will pull itself into given no outside disturbance. Surface energy continually pulls the molecules together, which naturally forces minimum area. Stretching out the liquid to a larger surface area directly opposes these forces, and the energy required for this deformation per unit area is the surface energy of that liquid. A classic example of a liquid with high surface energy is water, and even higher is mercury, and both show a characteristic 'beading' behavior when placed on a nominally clean surface. An excellent example of surface energy used in nature is the water strider, and insect which can walk on water so long as the mass it exerts on an area is less than the surface tension of the liquid. If the surface energy is exceeded, the surface will collapse.

Solids, unlike liquids, are not capable of smoothly changing their form. If a volume of water is removed from a sphere in zero gravity, the water will form itself into a smaller sphere almost instantaneously as it reconfigures into a new minimum-energy configuration. As solids cannot do this, solids usually have 'excess' energy on their surfaces when cleaved as the molecules on the surface are reactive but incapable of moving toward each other.

Given time, the molecules on the surface of a solid will react with anything available- usually oxygen in the air- and form lower energy, stable compounds on their surface. The quality of wetting, which is the ability of a liquid to maintain contact area with a solid, is directly affected by the energy available at their interface. It is for this reason that it is always suggested to expose new material on a surface when applying an adhesive- it provides more to adhere to.

Wetting relies on the tendency of all matter to move toward the lowest energy state available to it. The minimal system needed to understand wetting is a solid and a liquid brought together in a gas, and this is represented in Young's spreading equation. Young's equation states that if the energy across one unit of solid to gas interface is higher than the energy across one unit each of solid to liquid and liquid to gas interface, then the liquid will spread over the solid. This replaces an area of solid in contact with gas with an equal area of solid to liquid and liquid to gas interface, as a unit of liquid moves in between the two (Gans, 1972).

3.7 Wetting Revisited

Outside of the technical considerations involving surface energy, wetting is an intuitively accessible phenomenon. In nature one can see that certain surfaces such as rose petals and ducks are highly repellant to water and that the water tends to bead up and roll off of them. Soil and dogs, on the other hand, tend to absorb any water which comes into contact with them.

In terms of adhesives, wetting is a good measure of how much real contact is made between an adhesive and a surface. An adhesive joint with strong wetting is easy to imagine, with all voids in the two surfaces filled with adhesive. An adhesive joint with poor wetting can appear to have full adhesive coverage, but due to the lack of molecular attraction the adhesive to adherand interface can be of low area with air trapped under the adhesive rather than being forced out by wetting action and areas of adhesive which appear to be in complete contact but which are not actually in molecular contact.

The capillary effect, where a liquid pulls itself up the inside of a thin tube, is an excellent example of how wetting can cause an adhesive to improve its contact with a surface. The strong tendency for an adhesive to improve its area of contact with a wetted surface is also important for bond longevity, where ‘adhesive hysteresis’ takes place. In adhesive hysteresis, the close molecular bonds in an adhesive joint can reconfigure themselves and become stronger over time. In other words, the energy to separate the joint can actually become higher than the energy input to form the joint. This is a phenomenon unique to diffusive adhesion, adhesion by way of intermolecular forces. Strong wetting maximizes this effect by putting the largest possible amount of adhesive in range of the strong molecular attractions.

Wetting is measured in various ways, though all involve either measurement of the surface interface area of a solid with a given volume of liquid or the contact angle formed by the bottom of the liquid interface with the solid surface. Angular measurements are preferred in most cases because they are easier to measure accurately, and consecutive tests don't need to use the exact same volume of liquid each time (Gans, 1972). The contact angle can be split into two different measurements, the advancing and receding contact angles, and these are roughly approximated by measuring the contact angle of a puddle which is being added to and a puddle which is evaporating or having volume removed from it respectively. These classifications are shown in Figure 11, with the arrow up top showing the direction of liquid flow into or out of the drop and the angle being measured from horizontal facing the inside of the drop.

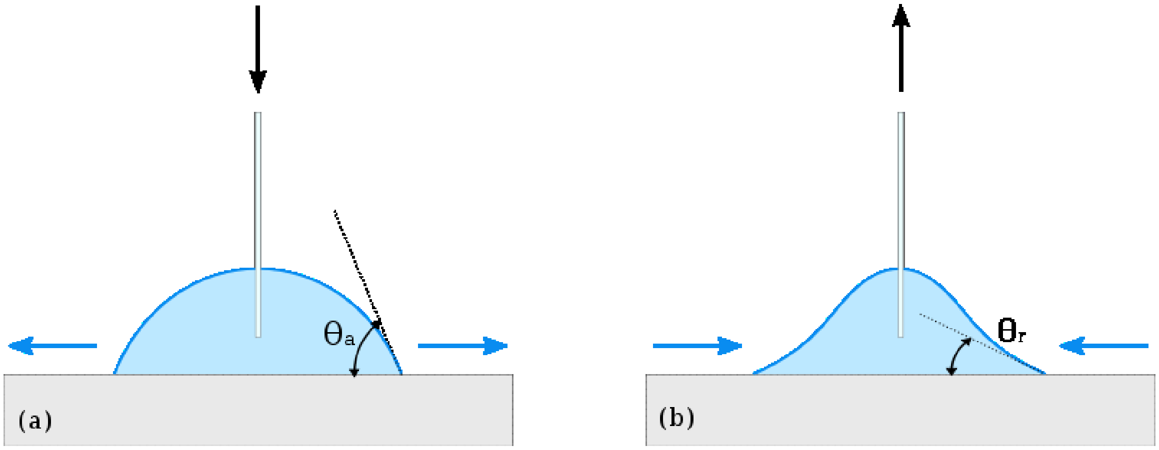

Figure 11 - Advancing and receding contact angles illustrated. Measurement is done visually using magnification and an optical angle measuring device such as a protractor,(Paumier, 2008)

3.8 Testing of Adhesive Joints:

The choice of testing methods depends on the bonding method used and the expected working conditions of the joint in question. In mechanical tests the samples are loaded to the point of failure. The nature of the failure-adhesive, cohesive, or substrate-is as important as the actual force required. Adhesive failure, in particular, can point to errors in adhesive choice or process. The three most common tests are shown in Figure 12.

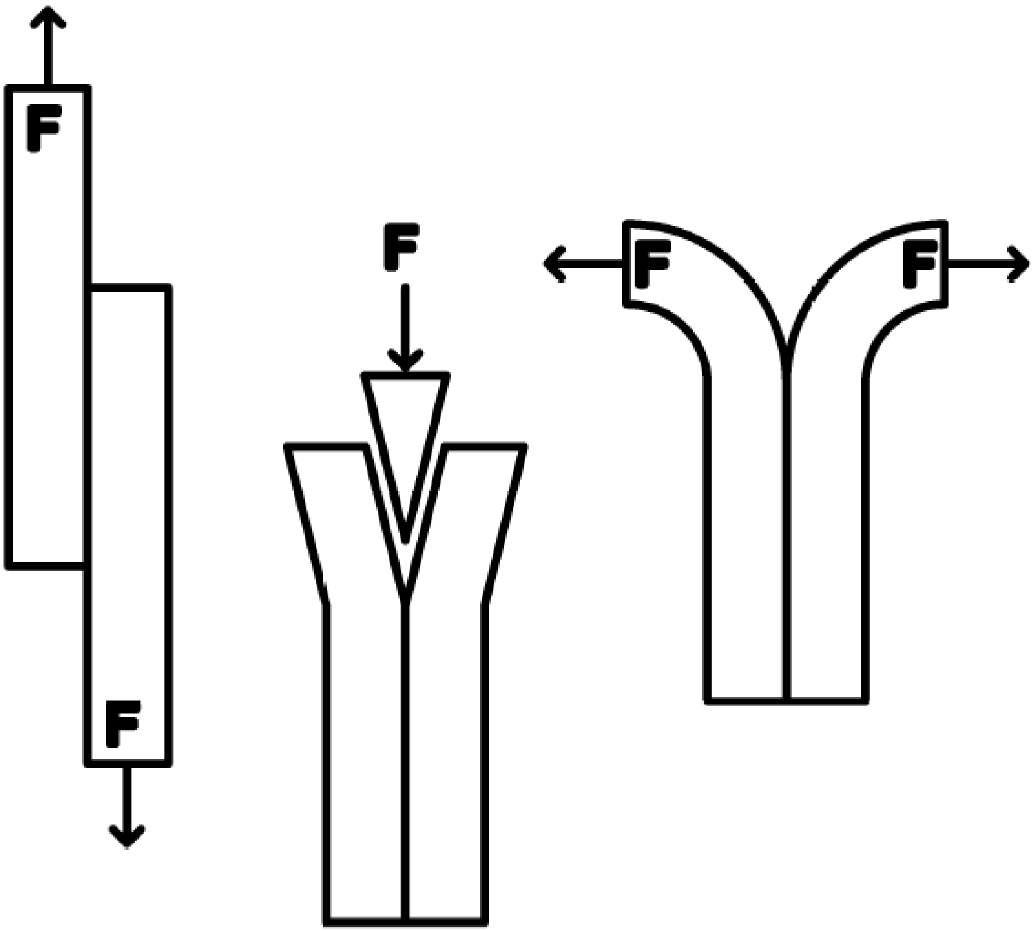

Figure 12 - Tests shown left to right: lap shear, wedge, peel

The lap shear test is the most commonly used test for medium to high strength bonds (The Adhesive and Sealant Council, 2010). A single lap joint is subjected to force in the direction of the bonded joint until failure. According to the DIN EN 1465 standard, the lap joint is to have an area 25mm wide and 12.5mm long in the direction of pull. The standard has the test carried out in such a way that fracture occurs in 45 to 85 seconds with the jaws of the apparatus moving at constant speed. The results are calculated in Newtons per square millimeter.

The wedge test involves driving a wedge between two sheets which have been bonded together. The wedge test can be used to measure the force of separation, though it is usually used to form a crack in the bond which is then subjected to environmental testing. By observing the progression of the crack after environmental testing, and further testing the strength of the bond, the long term performance of the bond can be predicted.

Peel tests involve applying force normal to the plane of the bond in order to separate the two bonded surfaces. The force required is recorded, and the distance between the moving clamps, during the entire process. This type of testing can provide relatively high resolution information about the adhesive and cohesive properties of the bond by combining analysis of the force/distance diagram with inspection of the broken bond.

There is also an active community of researchers doing work on adhesion of ice and physical properties. Due to its unique structure and variations in composition, ice is harder to characterize in a very general sense than many other materials of engineering concern. The physical properties can be quite different depending on the composition and solutes in the water, and the temperature and formation time of the ice.(Fukusako, 1990) The main driver in ice research is concern over icing on aircraft and standing structures such as power transmission lines and stations, and the danger caused by ice in collision with ocean going vessels. Fortunately, for many of these studies the focus is on the adhesive strength of ice as related to the materials bonded to and measures of surface energy, wetting, and roughness.

The results for the shear strength of solid ice are contentious, but an overview of different results by (Ashton, 1986) finds measured shear values in the range of 1500 – 2500 kPa for ice in the range of -5 to -20 degrees to be common among a number of different research groups. Of interest relative to these results is that (Haehnel, 2002) finds values of 1600 kPa to 3000 kPa for adhesion with extremely rough surfaces (slip plate, of the type applied to metal staircases to prevent slipping on ice) which might effectively be shear failure of the ice itself.

There are a number of papers on specific qualities of ice adhesion, but there are methodological differences that limit the usefulness of many of the papers in this context. The first researched paper on the relationship between wetting contact angle and ice adhesion angle formed the ice by precipitation of freezing drizzle on various surfaces (Dotan, Dodiuk, Laforte, & Kenig, 2009). While they did find a reasonable correlation between contact angle and adhesion strength, the method of ice formation was mentioned as being inconsistent between samples and there were small sample sizes with significant outliers in the data. A study conducted between researchers at Edwards Air Force Base and MIT (Meuler et al., 2010) mentioned the aforementioned method of depositing ice as possibly being inconsistent and chose to use columns of ice formed in tubes placed on top of sample specimens. The data from the MIT group was much more consistent, despite covering 22 different materials and doing over 220 combined trials, and is shown in table 2. The end conclusion was that there was a very straight-line correlation between the receding contact angle and the adhesive strength of ice over the entire set of measurements. The MIT group also noted the incidence of total adhesive failure versus partial cohesive failure of the ice layer in their findings and found that the incidence of cohesive failure could be as high as 78% in samples with high adhesion strength but did not occur at all with samples in the bottom half of adhesive strength. The MIT group also did tests at -5 and -15 Celsius to test against their nominal -10 degree tests and found a low probability that the adhesion values change significantly in this temperature range. Methodology in the MIT group involved 'casting' cylinders of ice onto steel plates, bare and with chemical coatings, in a freezer and then pushing the ice pillars off of the plates laterally using a load cell. The casting was done simply by placing hollow tubes on top of the plates and filling them with water. In terms of failure mode, the methodology is close to the current research. The formation of the ice into pillars prevents any extra conclusions on adhesion of thin layers of ice between substrates from being made.

Table 2 - MIT results on 22 materials. Notice that there is a linear correlation between adhesive strength and 1 + Cos(ϑrec) (Meuler et al., 2010)

substrate	θadv, water	θrec, water	no. of tests	fraction with complete adhesive failure	average strength of ice adhesion at -10 °C (kPa)*c*
bare steel	86.2 ± 3.3	25.8 ± 2.5	9	0.33	698 ± 112
PEMA	84.6 ± 2.4	68.0 ± 2.5	9	0.67	510 ± 101
99/1 PEMA/fluorodecyl POSS	97.5 ± 2.2	67.5 ± 2.2	9	0.22	475 ± 50
PMMA	83.6 ± 3.6	60.7 ± 1.3	11	0.73	463 ± 65
97/3 Tecnoflon/fluorodecyl POSS	127.0 ± 1.7	87.7 ± 4.8	11	0.82	412 ± 64
PC	93.4 ± 1.0	73.9 ± 3.3	7	0.86	400 ± 83
99/1 Tecnoflon/fluorodecyl POSS	125.7 ± 1.9	79.2 ± 3.4	13	0.92	392 ± 88
Tecnoflon	118.3 ± 1.4	73.7 ± 2.1	17	0.76	389 ± 63
PBMA	92.8 ± 2.4	74.6 ± 1.7	9	0.44	384 ± 52
97/3 PEMA/fluorodecyl POSS	105.4 ± 3.7	77.0 ± 4.7	8	1.00	367 ± 86
90/10 Tecnoflon/fluorodecyl POSS	126.6 ± 0.8	98.0 ± 5.3	9	1.00	345 ± 104
95/5 Tecnoflon/fluorodecyl POSS	126.6 ± 1.2	92.9 ± 4.3	15	1.00	328 ± 97
80/20 Tecnoflon/fluorodecyl POSS	126.0 ± 0.9	103.7 ± 4.3	11	1.00	313 ± 70
PDMS (Sylgard 184)	108.9 ± 1.5	91.7 ± 5.1	9	1.00	291 ± 44
95/5 PEMA/fluorodecyl POSS	122.2 ± 2.0	104.0 ± 5.3	8	1.00	278 ± 93
50/50 Tecnoflon/fluorodecyl POSS	128.3 ± 1.1	108.7 ± 3.4	8	1.00	265 ± 42
fluorodecyl POSS	137.6 ± 4.8	110.0 ± 3.8	15	1.00	250 ± 54
90/10 PEMA/fluorodecyl POSS	122.6 ± 2.1	107.6 ± 6.9	12	0.92	247 ± 45
70/30 Tecnoflon/fluorodecyl POSS	125.2 ± 0.8	110.0 ± 3.1	9	1.00	205 ± 40
50/50 PEMA/fluorodecyl POSS	125.0 ± 1.7	114.1 ± 2.4	8	1.00	185 ± 57
70/30 PEMA/fluorodecyl POSS	124.2 ± 0.9	116.4 ± 2.9	9	1.00	166 ± 44
80/20 PEMA/fluorodecyl POSS	123.8 ± 1.2	118.2 ± 2.4	7	1.00	165 ± 27

The underlying structure of the surface was shown to have a dramatic effect on the crystalline structure of the ice by (Archer & Gupta, 1998). The change in adhesion due to ice temperature was also thoroughly investigated. Between 263 and 253 Kelvin (-10 and -20 Celsius), the adhesive strength of the bond on unpolished aluminum stock dropped by over one third, though there was a much smaller drop of a further 5% by 233 Kelvin (-40 Celsius) which supports observations during the current research of the contraction of ice at low temperatures causing microfractures and reduced adhesion. Interesting to note is that the adhesive strength in the 253 to 233 (-20 to -40 Celsius) range was almost identical to the adhesive strengths of both polished aluminum and aluminum with hydrophobic coatings at 263 (-10 Celsius) degrees. Their fitted plot is shown in Figure 13.

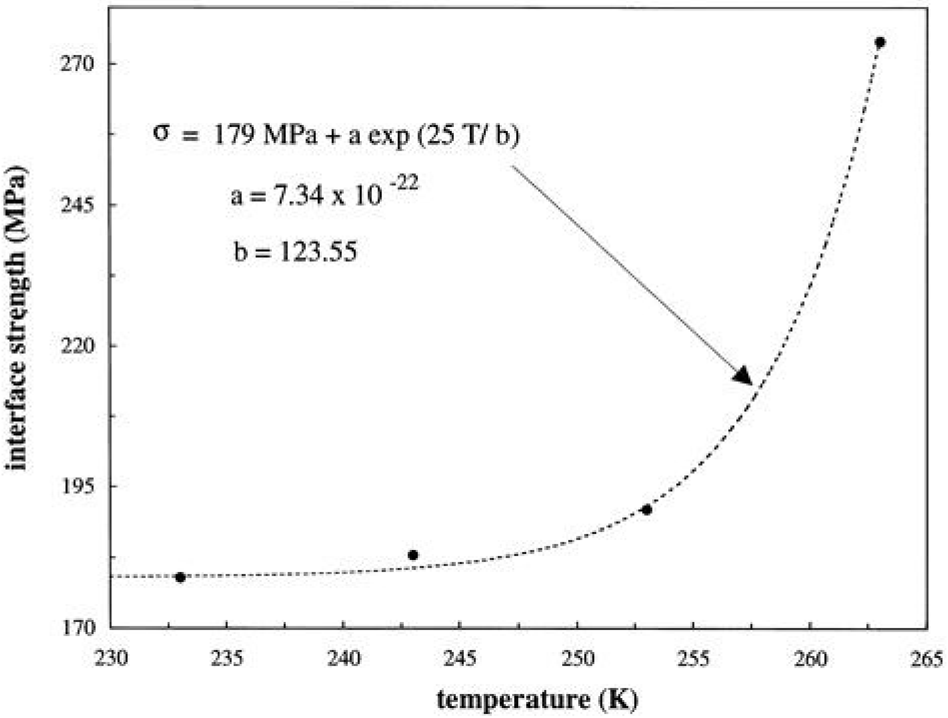

Figure 13 - Plot of shear strength versus temperature for polished aluminum, (Archer & Gupta, 1998)

Studies hoping to characterize the effect of surface roughness on ice adhesion were less successful, seemingly in large part due to decisions on methodology. In a test involving highly consistent ice samples cast in place on aluminum beams (F Hassan, P Lee, & P Lim, 2010), stress was applied to the samples by vibrating the beams and reading a strain gauge which was placed between the ice sample and the vibration source. The results had a very rough correlation when averaged, but were inconsistent even on the same sample with measured interfacial release forces varying by up to a factor of two between consecutive tests on each of the samples. Average results for ice adhesion to an 80 grit aluminum workpiece were close to 750 kPa, with results of under 200 kPa for test pieces lapped to 400 grit. A second study (Zou et al., 2011) used extremely high end equipment including multi-axis strain gauges and even electron microscopy in their testing, and conducting the tests in a 100% nitrogen atmosphere to control condensation. Tests were done using ice formed on lapped aluminum plates from drops of equal volumes of water. The area of a drop of liquid in contact with a surface can vary by very large factors- easily factors of 10 and more- based on contact angle, but this was not taken into account in the analysis as a major contributing factor. Since adhesive strength has a direct linear relation to surface contact area, this prevents any use of the data without very accurate information about the actual contact areas. These tests also found adhesion strengths very close to 750 kPa for rough workpieces and a decrease in adhesion strength of close to 50% for smoother workpieces.

Though there is little reliable information on correlation between roughness and adhesive strength, there are some papers which provide useful information in terms of upper bounds.(Haehnel, 2002) does a thorough comparison of ice adhesion to different substrates, in shear, and finds that on unprocessed aluminum the adhesion strength is in the range of 1500 kPa +/- 150. While these results are at odds for even the highest adhesions measured by Zou and Hassan, and certainly the results for smoother surfaces, it does match closely with the results of the current research. The methodology in that test, shown in Figure 14, is seen as particularly sound as it was highly repeatable. The test pieces were prepared by casting cylinders of test material in place using ice, then pushing them free with a load cell. The results were highly repeatable.

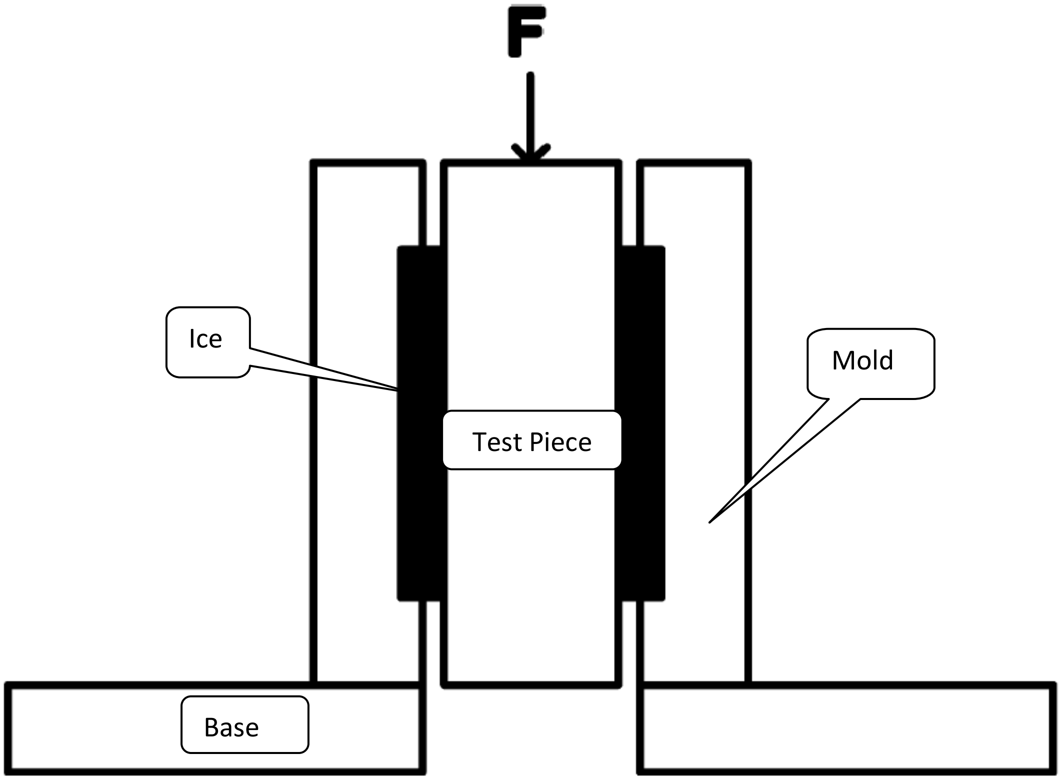

Figure 14 - Method of the US Army Core of Engineers. Ice is cast between test piece and mold, then the piece is pressed through with a load cell (Haehnel, 2002)

There are some specifications given for industrial products implementing the process of ice clamping, often referred to as freeze chucking. While there are a number of products on the market, they are very specialized products and by far the most prominent is the Ice Vice© series of freeze plates manufactured by Witte (Horst Witte, 2010). The only performance claims given in the company's material are a clamping force of up to 1400 kPa, clamping and unclamping times of 90 seconds, and repeatability up to plus or minus five microns. Working temperature of -6 to -10 Celsius is mentioned in press releases. No mention is made of materials used in the plates, nor their surface preparation. The Ice Vice© comes in a number of sizes, from roughly 2" x 2" up to 10" by 16". The apparatus used in the current research is 8" square, and clamps in less than 80 seconds.

Chapter 5 – Adhesive Apparatus Design : Applied Science and Automation

This chapter covers the design of the freeze chuck used in this research, and an overview of the technologies and construction methods. The design methodology for this device focuses on the usage environment, a CNC milling machine, as a starting point for making design decisions. The technologies used, thermoelectric coolers and water cooling, are covered in more detail as they were researched further to optimize their usage in this project.

5.1 – Choice of Operating Principle

There are three major designs of freeze chucks in industry, each with advantages and disadvantages in terms of construction and deployment. The performance of the different variations is quite different, as is their energy efficiency. Though it has by far the highest energy efficiency, conventional refrigeration was ruled out first due to the physical size of the system as well as the expense of custom made to order systems. Due to the necessity of dealing with refrigerants, it wasn't feasible to build a custom system using conventional refrigeration in house. The remaining two technologies, vortex tubes and thermoelectric cooling, were weighed on their advantages and disadvantages.

Vortex tubes have the lowest materials cost- a cooling plate based on vortex tubes is nothing more than a labyrinth plate attached to the cold air exhaust of one or more tubes. The tubes themselves are relatively inexpensive, as low as $100, and certainly much less expensive than conventional refrigeration units or thermoelectric cooling plates capable of reaching the same temperatures. On the other hand, vortex tubes use a very large volume of air and output only a fraction of the input air as cold air. The operating principle of vortex tubes is still debated, but a diagram of airflow through a tube and separation into hot and cold streams is shown in Figure 15. Air is pumped into a vortex generator at high pressure and room temperature, and the output of this generator is separated into hot and cold streams. A simplistic explanation of their function is that air in a vortex is separated into high and low pressure or fast and slow moving zones, and by the ideal gas law this implies that the slower moving air is colder than the faster moving air in a ratio more or less defined by their respective pressures. By controlling the fraction of air exiting the hot side with a regulator, the cold air fraction can be raised or lowered. Lowering the cold air fraction limits collection of cold air to the most central, slowest moving, lowest pressure, and coolest air. The amount of air required to keep even a small plate of six square inches below freezing kept a five horsepower compressor running continuously during testing, which is expensive both in terms of energy input and noise output. This quickly disqualified vortex tubes from the full scale apparatus. 1800 watts of cooling from a vortex tube requires a 13,400 watt compressor (Newman Tools Inc, 2009).

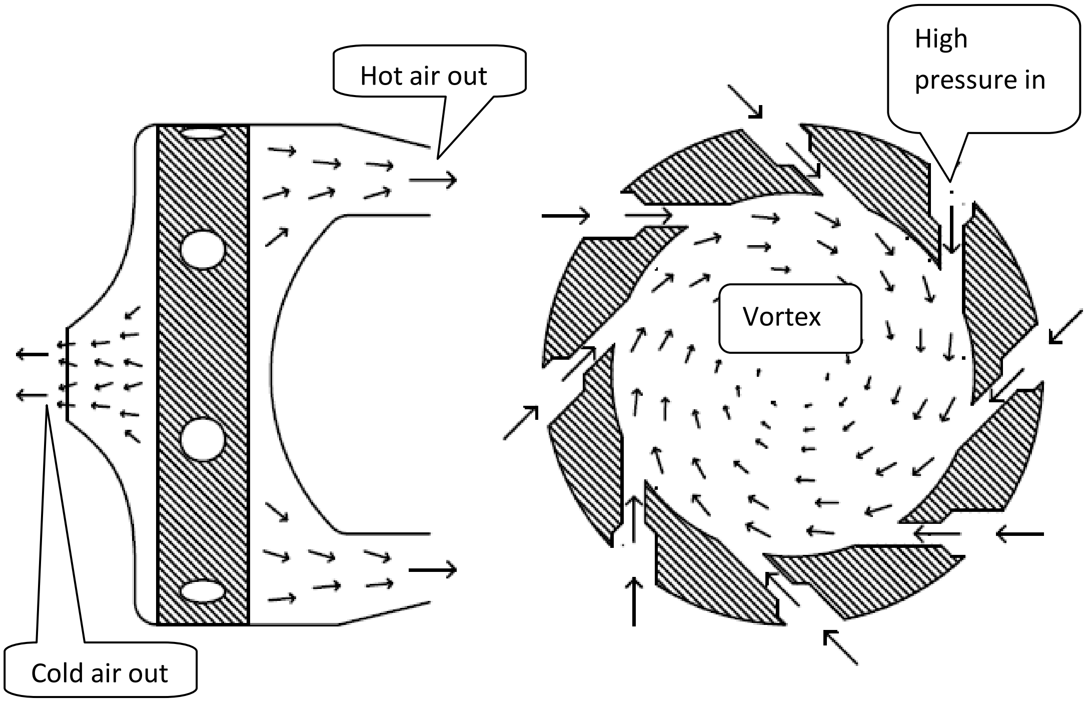

Figure 15 - Vortex tube. Side view on left shows exhausts and vortex generator, front view on right shows only vortex generator. Vortex tubes vary from ½" to 2" in diameter.

Thermoelectric coolers (TECs) or ‘Peltiers’, named after the physicist who discovered the effect on which they rely, are electrically operated heat pumps capable of moving a large amount of heat a small distance. A typical TEC is shown in Figure 16. TECs require a large amount of electrical current and active cooling on their hot sides, but are capable of higher performance then the other options at a much lower total operating cost. Construction complexity is substantially higher on a system using TECs, but given that the author is a machinist this wasn’t a strong point against their use. TECs require roughly one watt of power for each watt of cooling, but must have their hot sides actively cooled for twice as many watts of heat as they are pumping.

Once operating principle had been decided on, the design of the apparatus follows logically as shown in Figure 17. TECs are used to cool a top plate, and a water block is used to cool the TECs.

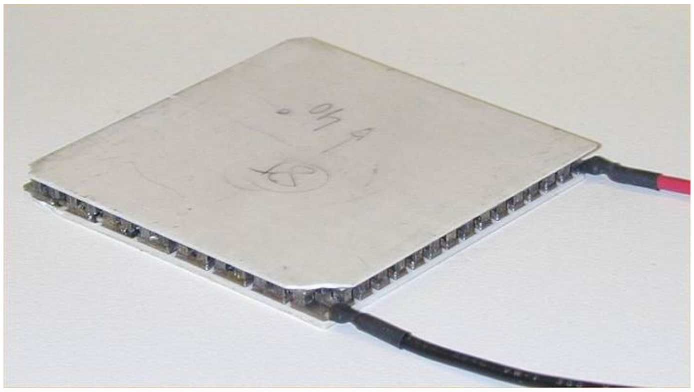

Figure 16 - A TEC, unsealed and showing thermocouple pairs. This unit is roughly 1.5” square, (Wikimedia Commons (public domain))

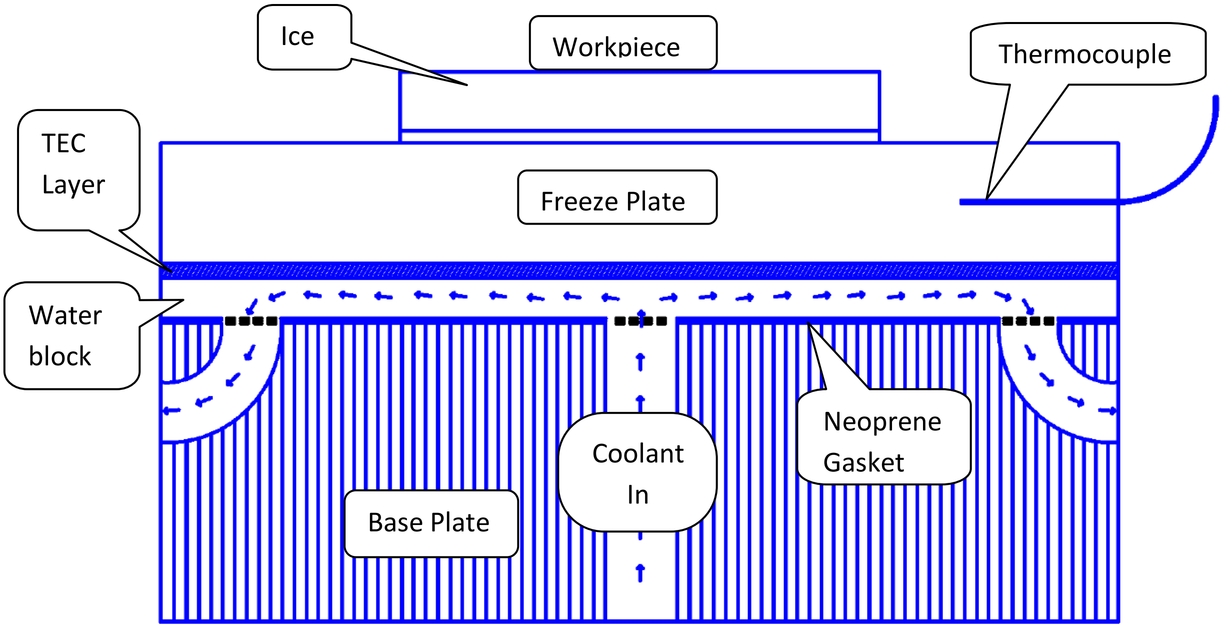

Figure 17 – Cross section of the apparatus design. Apparatus is 8" wide and 4" tall. Coolant flow is simplified here, but shown in greater detail in figure Figure 21

To get an idea of the basic performance expected, it is useful to do a simple thermal calculation. The top plate of the apparatus is eight inches square and one inch thick, with a total volume of 64 cubic inches. An aluminum workpiece the same size as the top plate but 1/8" thick, and a layer of water 1/64" thick are assumed. In general, workpieces are much smaller than the top plate and the layer of water beneath them is much thinner.

Calculations are shown in table 3 below. Units are standard, with temperature delta based on a 20 to -10 Celsius temperature change. The heat of fusion for water is expressed over a delta of one as it is only applied once. Thermal losses through the top plate are assumed to be negligible compared to cooling power, and decrease in TEC efficiency isn't accounted for as the TECs used are overpowered.

Assuming a TEC array with a cooling power of 1200W, it is shown that the cooling plate will reach operating temperature in approximately 78 seconds. As roughly ¾ of the energy is used to cool the freeze plate itself, a thinner top plate could be implemented on a proven design to drastically reduce wait time.

Table 3 - Thermal calculations, device as tested

Component Material	Specific heat per cubic inch	Volume	Joules per degree for component	Temp. Delta	Energy
	J/(in^3*°C)	in^3	J/°C	°C	Joules
Aluminum	40	64	2549	30	76455
Water	69	1	69	20	1372
Workpiece	40	8	319	30	9557
Ice fusion	5473	1	5473	1	5473
Ice	32	1.083	34	10	345

Total energy (J)	93202
Seconds to operating temp.	77.67

Available cooling power (Watts)	1200

5.2 TECs in Detail:

A thermocouple, commonly used for sensing temperature, is a pair of wires connected together at one end and with voltage measured across their other ends. The working principle of a thermocouple, the Seebeck effect, states that a conductor subjected to a thermal gradient will generate a voltage along that gradient. Like most electromagnetic phenomenon, such as the operating principles of motors/generators and piezoelectric materials, the Seebeck effect can work in both directions and thus if a current is applied to a thermocouple then a temperature change will be effected. The effects of current, and the reversibility, are attributed to Peltier which is why TECs are called Peltiers and not Seebecks. A TEC is simply an array of thermocouples sandwiched between two ceramic disks, the disks being there for structural support and additionally for electrical insulation with minimal thermal resistance. A TEC capable of carrying 400W of heat is only two inches square and just over 1/8” thick.

TECs are limited in terms of wattage they can pump, which is mainly governed by surface area once out of the micro scale devices, and also the maximum temperature gradient possible across the device. Most single stage devices are capable of a 70 +/- 2 degree gradient (TEC Microsystems Gmbh, 2011). To affect larger temperature gradients between the heat sink on the hot side and the cold side of a TEC, they can be stacked to make compound TECs. Each layer must be capable of fully absorbing the heat thrown off by all previous layers, so maximum cooling power is decreased. Two, three, and four stage TECs can typically provide temperature gradients of 100, 115, and 125 degrees respectively. A four stage TEC with the same footprint as a single stage TEC can only move 4% as much heat, but across a much larger gradient. As performance data shows, a TEC becomes less effective as a heat pump the closer it is to its maximum temperature delta.

TECs are quite durable in use, but care must be taken to ensure good thermal contact between the TEC and the heat sink as TEC lifespan can be seriously decreased by the development of hot spots on the surface. They are relatively low voltage devices, with the aforementioned 400W device running on only 12 volts, thus requiring high current power supplies for operation. Science is not for the faint of heart, so the test apparatus uses four 550W TECS as those are the largest which could be found. The performance of a TEC, in heat transfer ability, degrades as the delta gets larger. The degradation is essentially linear, and the chart in Figure 18 is typical of TEC performance. Note that Figure 18 is a performance chart for a third party TEC based cold plate and is used as a reference to display the linear reduction of cooling power with temperature delta rather than performance specifications of the current apparatus.

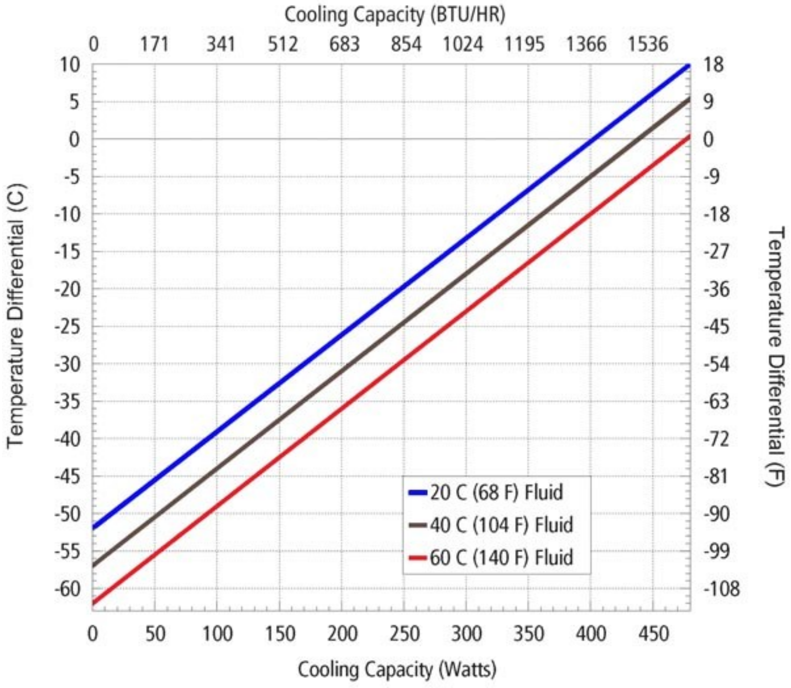

Figure 18 - TEC performance, Thermoelectric Cooling America Corporation

5.3 Heat Sink Design:

At full capacity, the combined power of four 550W TECs could be pushing out more than 2200W of heat. The lowest temperature which can be reached is relative to the temperature of the hot side of a TEC, and a TEC becomes less efficient the closer it is to that maximum delta. Therefore the cooling system should ideally have enough capacity to not only remove all heat produced, but enough to do so while maintaining an equilibrium temperature at the hot side which is as low as possible. To minimize the size of cooling equipment which had to be directly coupled to the workholding device, liquid cooling was an obvious choice.

Parkinson's law states that work expands so as to fill the time allotted for its completion. Similarly, if looking for the cutting edge in engineering it would seem to be wisest to watch those for whom an acceptable level of peak performance has not been defined. A large research community to verify performance results, and a free market where success and financial incentive are based solely on those results, would be of immense benefit in pushing technology forward. Millions of computer overclockers, users wishing to run their computers faster than their rated specifications, form a demand for high performance liquid and air cooling equipment in small form factors which is unprecedented anywhere else in industry. Users often construct elaborate and high performance systems, and are obsessed with benchmarks and thermal performance measurements.

Observing the evolution of PC water block design, along with the empirical data provided by the tests, provides an invaluable practical overview of different designs. While the science to back up even the most modern designs is quite mature, the proof of design is in implementation which had not generally occurred at that time.

Initially, surface areas combined with flow rate were considered to be the most important factors in water block construction and labyrinth style blocks based on this design are still common in industry, a typical current labyrinth style block is shown in Figure 19. Turbulence, even at the cost of flow restriction, was shown to be a major determining factor in water block efficiency (Procooling.com, 2005). It is commonly known by engineers that laminar flow can form a slow moving layer at boundaries, limiting heat transfer, but the effectiveness of a much slower turbulent flow versus a much faster laminar flow had to be borne out empirically to gain acceptance in the PC cooling world. Hobbyists, and commercial providers, had been working on designs to optimize turbulence at least as early as 2003(Madshrimps.be, 2003), and the labyrinth design evolved into pin arrays and labyrinths with wavy walls and floors optimized for turbulence. It was in the period from 2003 to 2005 that commercial units started producing water blocks that induced a turbulent flow on entry to the block by forcing the water through a series of slits or holes onto an array of pins or grooves as in Figure 20, and the performance of these allowed them to completely overtake the market in a short span of time (Nelson, 2006). In 2006 and beyond this technology is used in nearly all PC water blocks. As of 2010, the top performing blocks use either extremely tight pin arrays or very thin long channels, along with a variety of ‘jet plates’ to apply turbulence at entrance of the water(22 waterblocks tested - roundup, 2010).

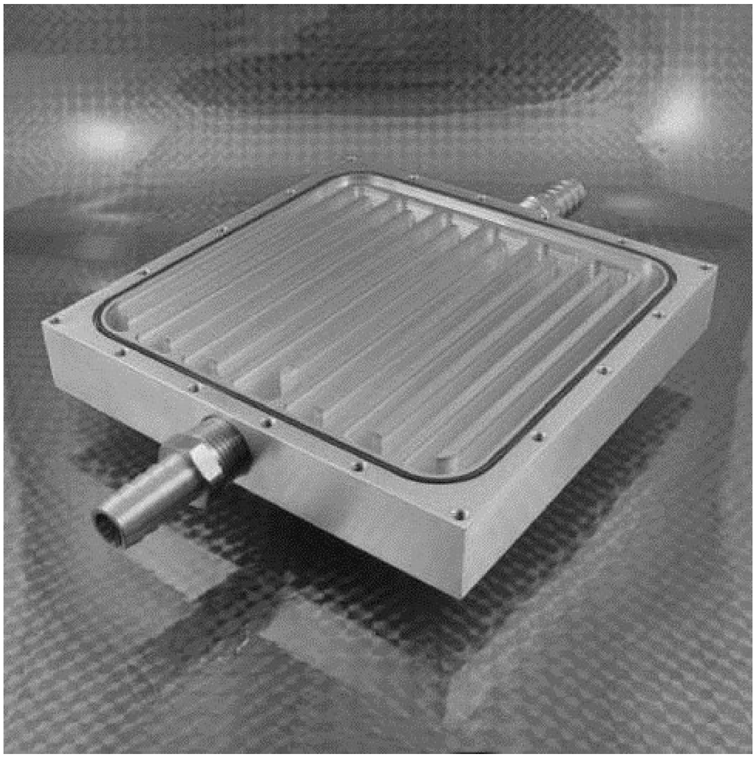

Figure 19 - Standard industrial water block design, simple labyrinth. Block is 4” square. (Custom Thermoelectric, 2011)

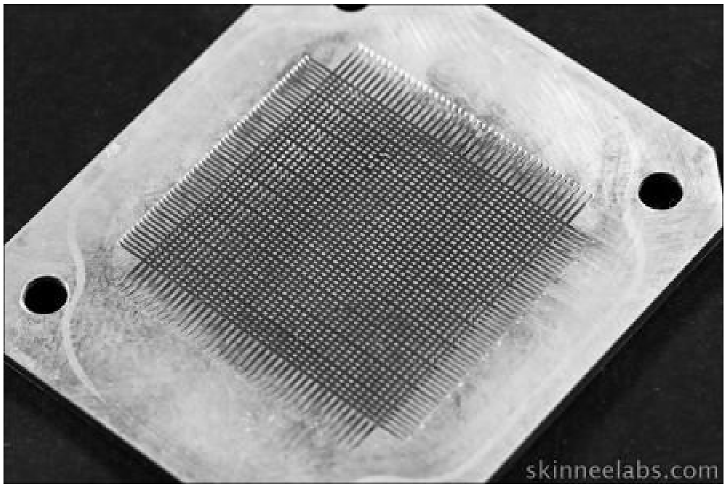

Figure 20 - Modern pin array, jet plate not shown. Cut area in center is roughly 1" square (Skinnee Labs, 2011)

Another area hotly debated in PC cooling circles is the coupling of heat sinks to the chips they are being used to cool. Thermal interface materials can be quite expensive and products accepted as being of high quality start at the low end with gels containing majority percentages of silver dust up to compounds made from diamond dust. It is notable that in tests there is not a very large performance difference between the best and worst thermal compounds (Rutter, 2011), and that improving the quality and flatness of both mating surfaces is of even greater importance. The simplest proof is that the best thermal compounds have conductivity less than 2% that of metallic copper, the material used in most heat sinks, making the smallest defect in fitment substantially more important than the compound used to fill it. The cost of using one of the best thermal compounds (Arctic Silver 5, $100) and lapping all mating components was still low compared to the total time invested in construction of the water block and so both were done.

The water block was intentionally overbuilt as optimization of water block designs is a very advanced simulation problem, and exceeding the requirements of the TECs allows the research to proceed under the assumption that they are always being maximally cooled and thus their performance should be constant relative to incoming coolant temperature. This was verified by noting that the outgoing water temperature was not noticeably lower than the incoming water temperature. Water comes into the block at ground water pressure and full flow rate.

5.4 Apparatus Design and Construction:

The apparatus was constructed out of commodity materials and components, though value was added through machining and modification of those materials and parts. The first components sourced were the TECs, which measure 62.5mm on an edge and are rated for 550W. As seen in Figure 17, from the top the construction is a top plate, four TECs, a water block, a neoprene gasket, and a base block. The entire sandwich is held together by screws which bear on the top plate and screw into the base block but which do not contact any of the other components (clearance holes).

The water block used to cool the TECs has a number of differences when compared to the PC water blocks which were studied. The goal in designing the water block was to take as many lessons from them as possible while taking the much larger size and structural requirements of the TEC block into account. Maximizing thermal contact over a small area is usually a simple matter of lapping both components until they are quite smooth, but over a large area it is much more difficult to have any piece of metal remain flat even if it is ground or machined flat originally. To this end, good thermal contact was ensured by using a large number of fasteners tightened to roughly equal torque over the surface of the apparatus, seen in Figure 25.

After sufficient material was designed in to allow for the fasteners, it became clear that liquid flow would have to be split in four directions to ensure a roughly uniform thermal gradient across the four TECs. Fluid flow is shown in Figure 21. The last concern was that the area of the block might allow flow behavior causing ‘dead areas’ of slow liquid throughput and so the choice was made to limit the total channel cross sectional area between the inlet and the outlet to four times the cross sectional area of the inlet hose. All these design choices were informed by the manufacturability of the water block by

CNC milling. Most if not all of the high performance PC water blocks are fabricated by either casting or saw slitting, with all current top performers appearing to be saw slit models, and so channel sizes had to be greatly enlarged. The chosen design, shown in Figure 22, minimizes milling time and cost, which are both large as copper is slow to machine and fond of entangling and breaking cutters. The design aims for a uniform distance from inlet to outlet for water across the plate area, and increases turbulence and surface area through use of irregular walls and in-channel posts.

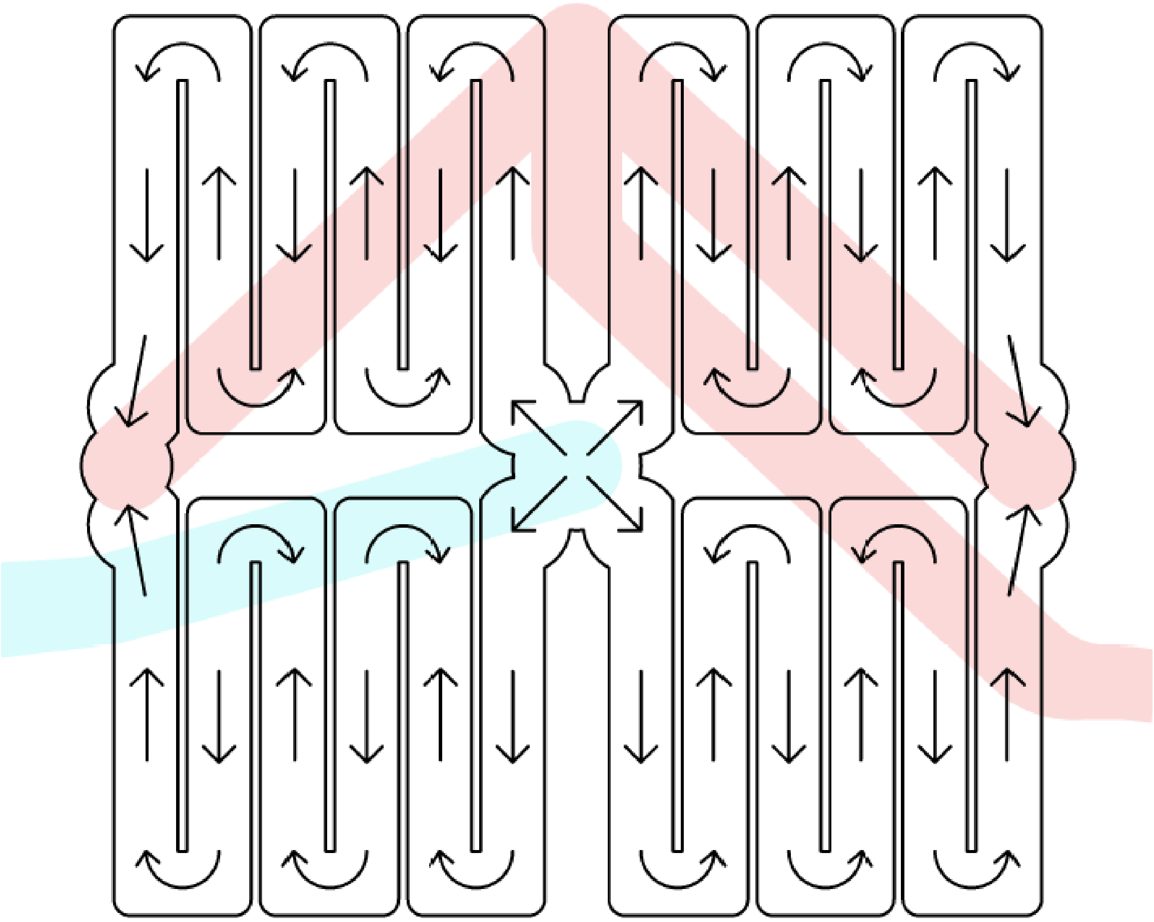

Figure 21 – Coolant flow through water block. Coolant comes in to a line below the block (blue), is distributed through the block (lines), and exits through more plumbing (red). The exit is designed to keep all paths through the block the same length.

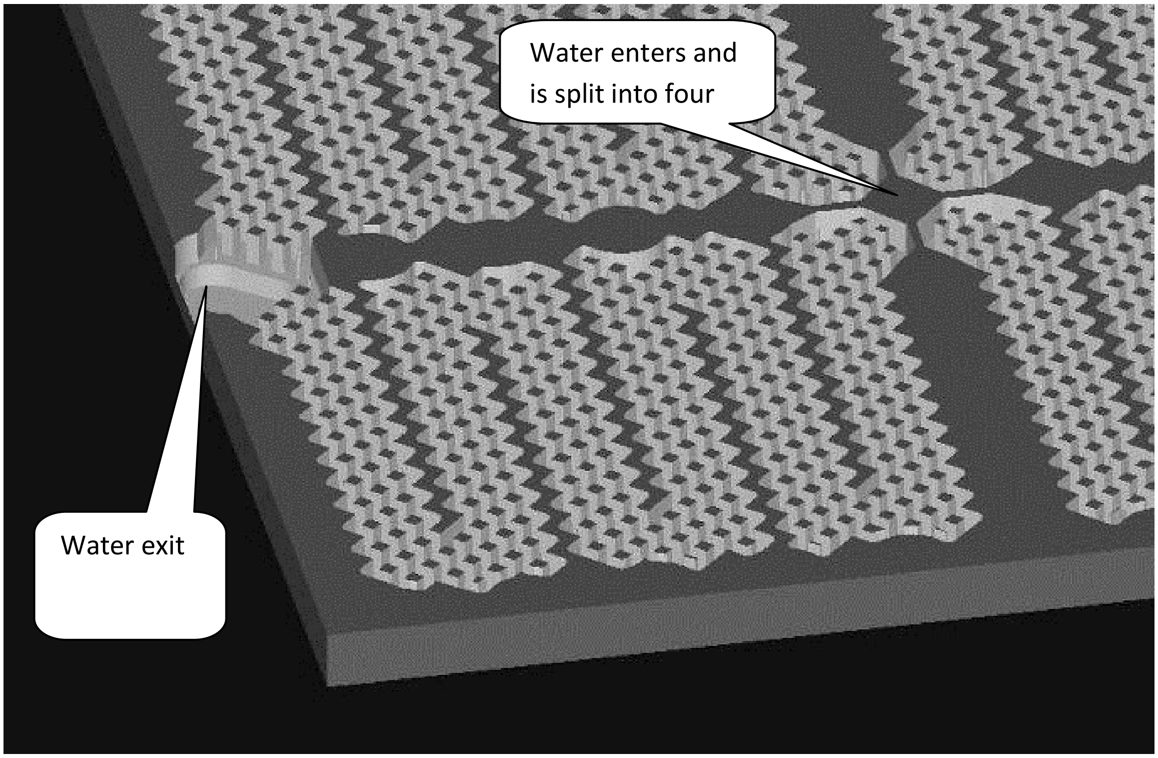

Figure 22 - Close up rendering of one quadrant of water block. Pins are roughly 0.1" on a side and channels are nominally 0.5" wide. Depth of cuts on the block is 0.375" and the block is 0.5" thick. Face shown is on bottom in use and water is pumped in from below, flowing between the block and the neoprene gasket.

The base and top plate of the freeze chuck are constructed from 6061-T6 aluminum. The base contains only channels for water and tapped holes to hold the top plate on. It is of note that the copper water block has through holes for, but does not come into contact with, the screws holding the top plate to the base plate. There is a neoprene rubber gasket between the base plate and the water block. Though it will still occur over time, ensuring that there is no metal on metal contact between the copper parts and any of the aluminum parts will substantially slow the process of galvanic corrosion. The use of fresh water, which is a poor electrolyte, further aids in prevention of corrosion.

The TECs are powered by two 1000W ATX power supplies, shown in Figure 23 and Figure 24, each connected to two of the TECs. The temperature of the system is controlled by an Omron PID temperature controller attached to a thermocouple embedded in the top plate. To avoid overloading the power supplies, the controller is set to a maximum frequency of 1 Hz but this has turned out to be unnecessary as hysteresis caused by the thickness of the top plate has the temperature moving down 1-2 degrees further after the TECs are turned off each time.

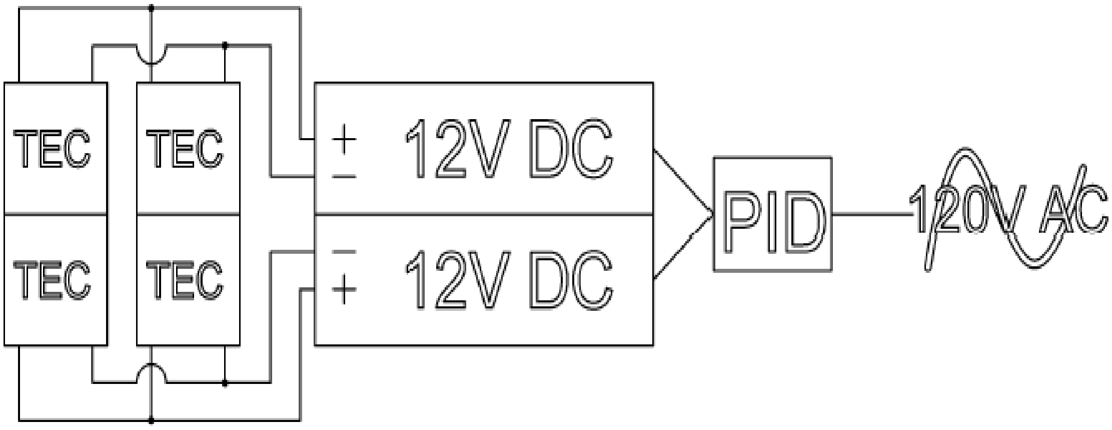

Figure 23 – Electrical Diagram showing PID control feeding two ATC power supplies, which in turn feed two TECs each. Each ATX power supply outputs 50-60 amps in use, split between two TECs

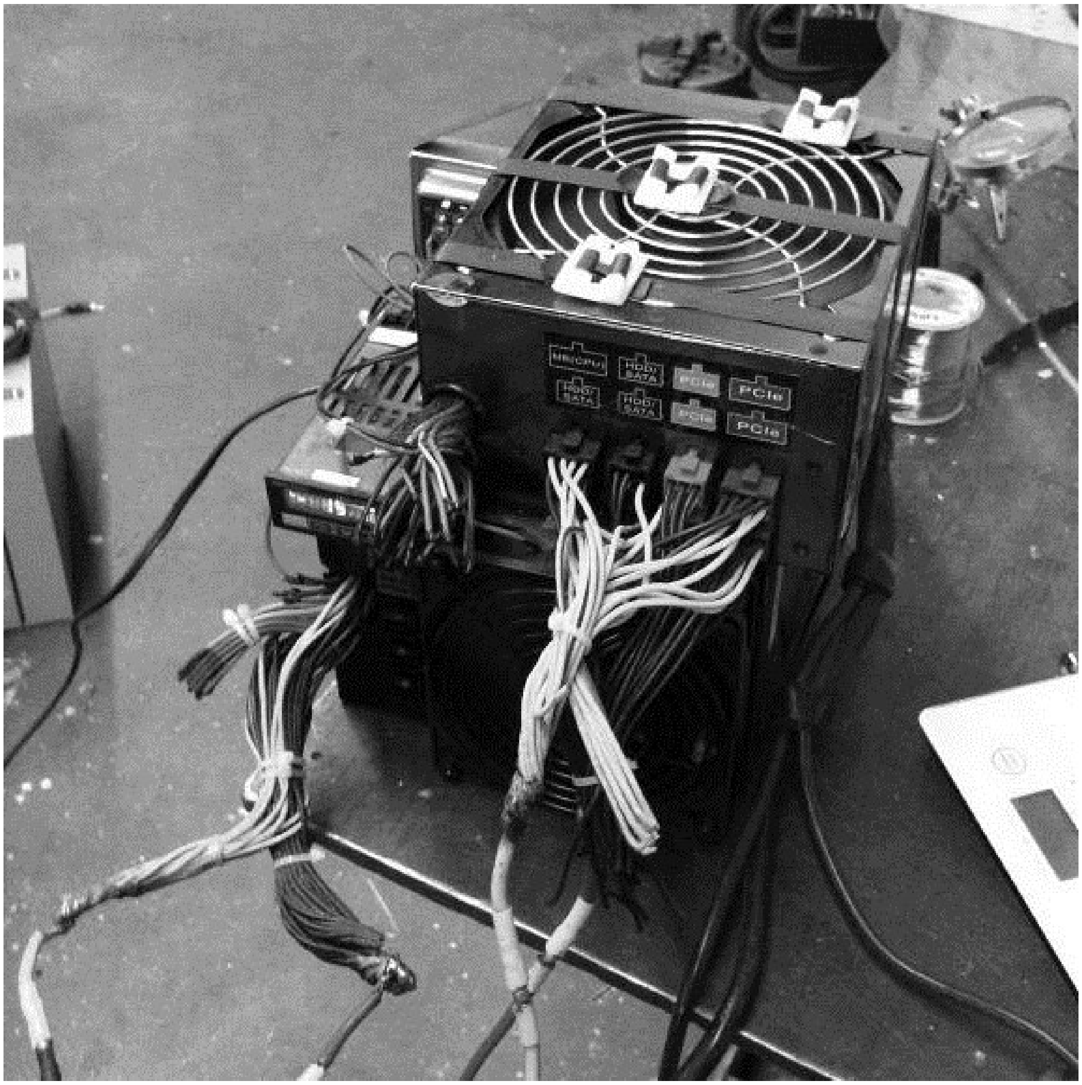

Figure 24 - ATX Power supplies stacked, PID control on left

Older ATX power supplies can only supply a fraction of their maximum wattage at a particular voltage, but newer high end supplies designed for usage in multiple supply systems are designed to provide higher percentages of their total wattage at 12V as this is the voltage used by high end graphics hardware. The two power supplies chosen are both capable of outputting their entire rated wattage at 12 volts. The TECs are rated at 550W at 16 volts, and are derated to roughly 300W each at 12 volts.

5.5 Performance Specifications and Basic Tests:

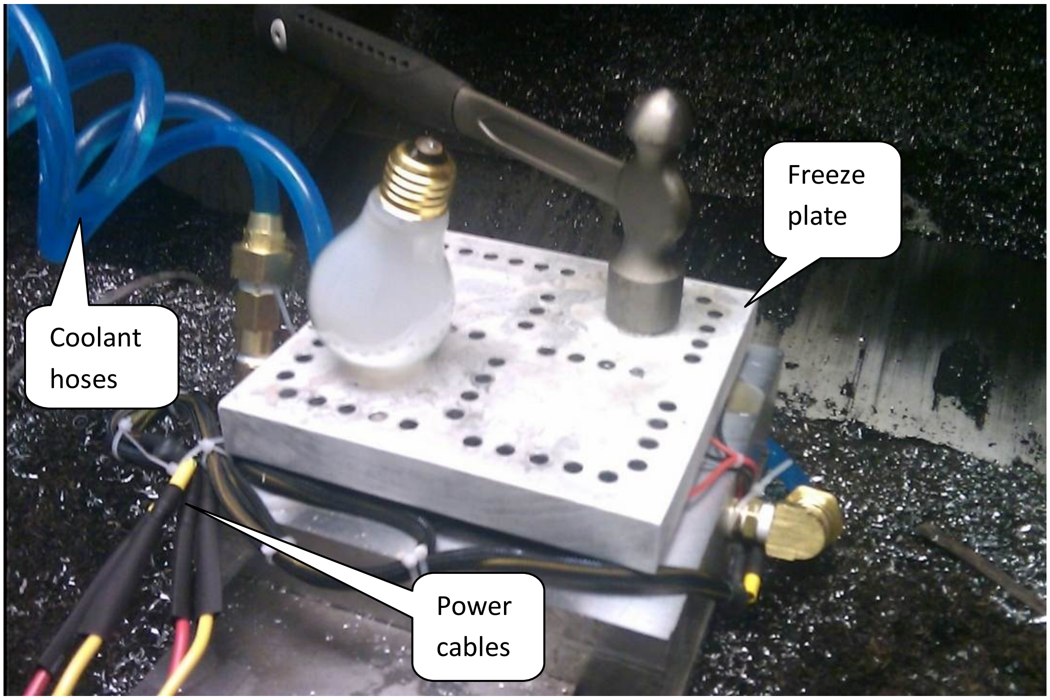

Figure 25 - Apparatus during initial tests holding a hammer and light bulb securely

Initial testing of the block was performed to determine basic operating parameters and to verify reliable operation, with the second run being used to try holding some odd objects as seen in Figure 25. Water for the cooling plate was supplied directly from ground water, and comes in at roughly six to ten degrees Celsius depending on the time of year. To test the difference between the measured temperature of the thermocouple embedded in the top plate and the true surface temperature, a piece of black paper is frozen to the surface of the plate and measured with an IR thermometer. In general, the top plate reading is roughly seven degrees higher than the reading of the thermocouple in the range from 15 to -60 Celsius. For this reason, a setting of -20 was chosen for the temperature control which results in an interface temperature of -11 to -13 degrees. This is in the range of roughly constant ice adhesion strength from -5 to -15 mentioned in the literature, but being at the lower end it insures that the tops of large pieces can also reach at least subzero temperatures.

Current consumption of the TECs was measured with a clamp DC ammeter on the wires between the power supplies to the TECs. During operation each line measures at between 52 and 60 amps of current, and each power supply feeds two TECs, so each TEC pulls 26-30 amps of current. 12V is relatively safe, even with water involved, but as an added layer of protection ATX power supplies have excellent fault mechanisms for shorts and large current spikes.

Peak current consumption of 120 amps at 12 volts results in waste heat of 1440 watts on top of heat pumped, and so at maximum cooling the water block must deal with roughly 2640 watts of heat dissipation. To stress test the apparatus, it was turned on with a 1500 watt heat gun trained on the center of the freeze plate. The heat gun was at maximum settings (550 degrees Celsius, 300 CFM of air) and held 2" above the plate. The plate succeeded in reaching freezing temperatures despite the large thermal load and even frosted over.

The minimum attainable temperature of the device was tested by leaving it turned on at full power for one hour. At the end of the hour, the plate had completely covered with 1/4" of solid frost condensed from the ambient air and the internal thermocouple read negative 61 degrees Celsius. Based on ground water temperatures of six to ten degrees, this verifies that the TECs were operating at better than 90% of their theoretical capacity. Unfortunately, a camera was not available at the time of this test, but all in attendance agreed it was really, really cool.

5.6 Testing Rig:

The setup for testing the holding power of the apparatus went through a number of design revisions. Most of the revisions were due to advances in optimizing the holding power of the setup, as preparation was determined to be the major contributing factor in adhesion strength. The initial testing setup consisted of a 25mm pneumatic cylinder which was attached firmly to a workbench and the test piece by ropes and then tensioned up until the pieces detached. Aside from the excitement of test pieces being propelled across the room at high speed by the tension in the rope, the cylinder turned out to be underpowered for some pieces which added the excitement of not knowing when a piece might release. Though science is an endeavor for the brave, the line between bravery and foolhardiness is to be respected in experimental design.

The following three test setups used two dual 20mm air cylinders first to push the pieces off in shear and then to pull the pieces off vertically in tension. The tension testing setups were designed to test the adhesion strength of the pieces without ice formed by the shallow puddle of water surrounding the workpiece affecting the results. As it turned out, the adhesive force may be even stronger in tension than in shear, and a 240lb (1650 kPa) vertical force was insufficient to remove even a one square inch test piece. Smaller area test pieces were tried, but smaller areas lead to more errors in placement and proper squeeze out of the water under the piece and thus larger swings in experimental readings.

The final test setup uses an 80mm pneumatic cylinder capable of pulling 1050lbs at maximum pressure. A diagram of the final setup is shown in Figure 26, with a photo in Figure 27. Chains rated for a 1400lb load are TIG welded to the nut on the cylinder rod, and a long 1/4 -20 nut is welded on to the opposite end to allow attachment of test pieces. Aluminum test pieces are cross drilled to allow a ¼” grade 5 bolt to be passed through them, but other test pieces are held in a small steel cage which is designed to attach the same way.

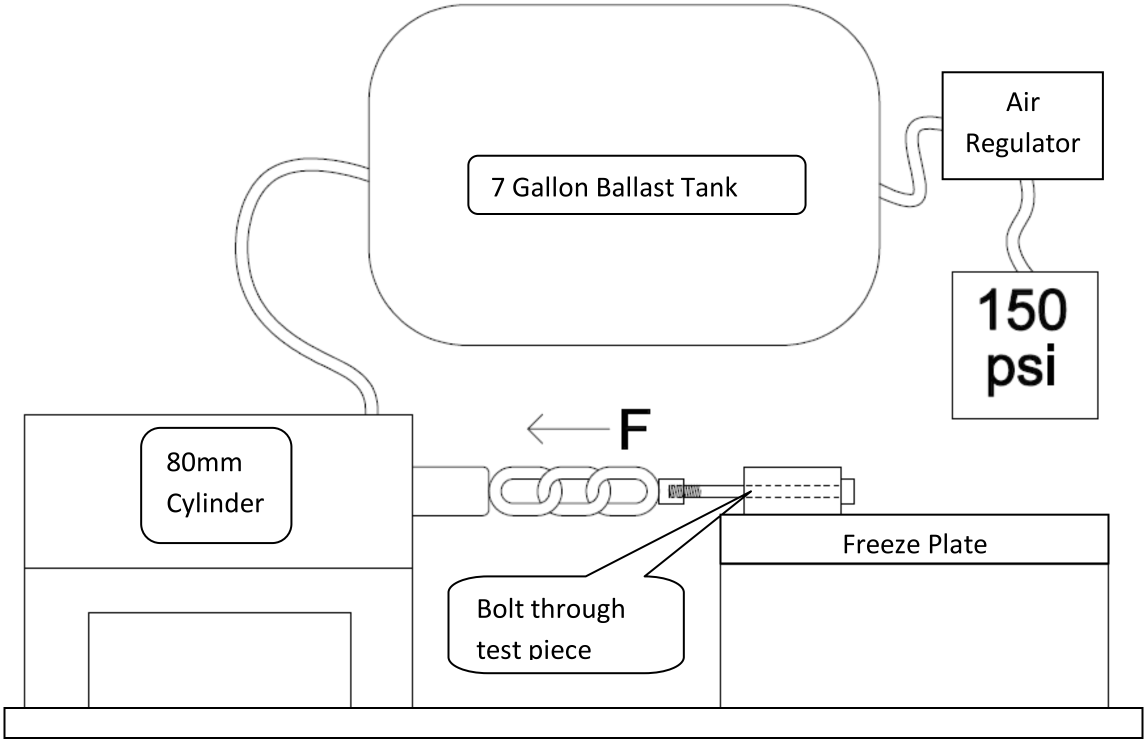

Figure 26 - Test rig diagram showing connections

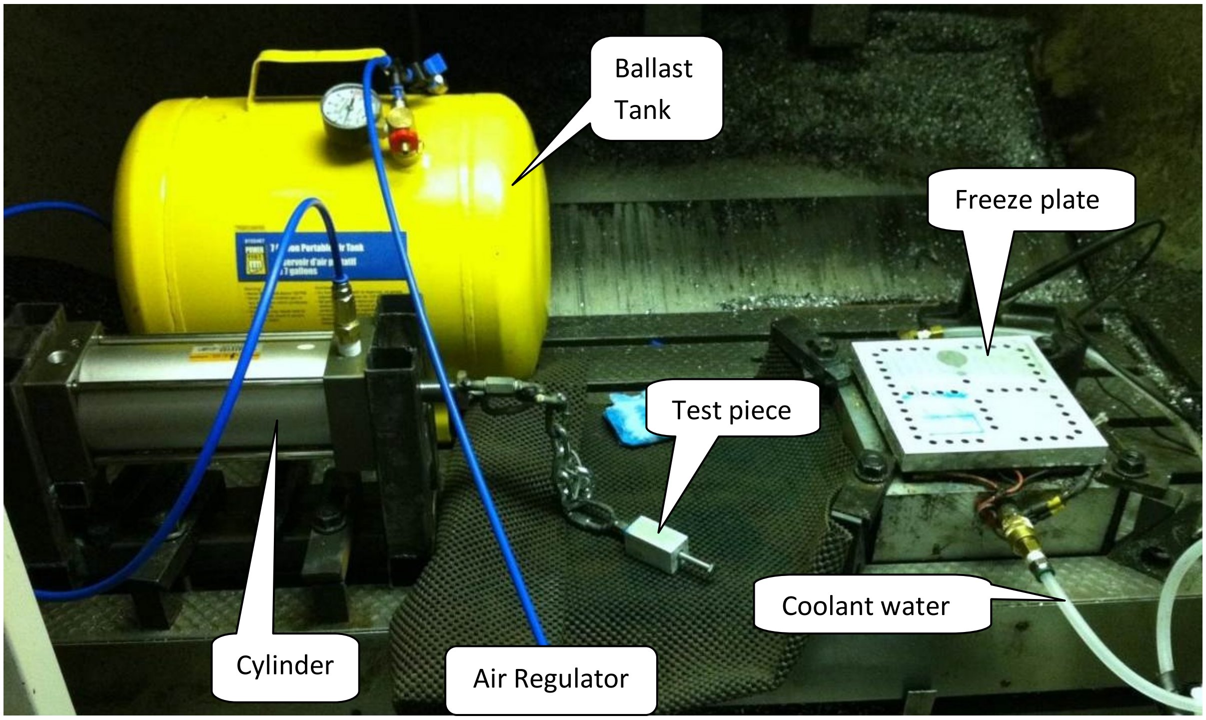

Figure 27 - Full test setup, air cylinder and ballast on left and plate on right.

Air is fed to the cylinder using a tire inflation device with digital pressure readout. Unlike round gauges which only read accurately above a certain fraction of their range, digital gauges read down to single digit pressures. To insure against pressure spikes which would result in false readings, the total air volume of the system was increased by inserting a ballast tank between the inflation device and the cylinder. The ballast has sufficient volume compared to the stroke volume of the cylinder that the pressure of the system changes by less than one psi when the cylinder is collapsed. Air is pumped into the ballast at one end and exits to the cylinder at the other. The lines from the ballast to the cylinder and to the gauge are of equal length and diameter to prevent constriction from causing false readings.

For safety reasons, the tests were conducted inside a vertical machining center (VMC). Though the VMC wasn't used specifically for any tests except cutting trials, it did provide a sturdy surface to which clamps could be applied and there are clear polycarbonate windows on the front doors which can be closed during testing. Polycarbonate is also known by the trade name Lexan and the colloquial name 'bullet proof glass'. The author has personally witnessed some large, fast travelling objects bouncing off those windows. The final tests are set up such that the cylinder only uses the last inch of its travel to remove the test pieces, thus they release with relatively low velocity. This also aids with prevention of mars and dents on test pieces which compromise flatness and repeatability, as the predictable landing area was padded with soft foam. A heat gun was used to thaw the plate between tests as the time for the plate to reach room temperature from frozen is substantial, over five minutes.

The water used in testing was distilled water which had been dyed blue with aniline dye. The dye is very strong, and a very small amount provides a very dark color, so it is believed that the dye would have very minimal effect on the freezing temperature of the water. The dye has two main advantages: the first is that it is almost impossible to see the water or ice in photos without the contrast, but the more important advantage is that the dye allows analysis of the bond failure. This turned out to be absolutely critical in interpreting the test results.

Chapter 6 - Time To Try The Tests

The original plan for testing the apparatus was to test various materials and compare their relative shear release points. However, during testing with the first material, aluminum, it was noted that preparation of the material has a substantial effect on adhesion strength. This suggests that small changes in procedure or sample preparation can have large effects on holding forces, in fact larger effects than the differences between most materials. Due to these initial findings, testing was done primarily with aluminum test pieces to observe the effects of part preparation on the test results.

6.1 Early Observations

A phenomenon which has been named 'ice damming' was noticed early in testing, and is pictured in Figure 28. It was noted that the fillet of free ice which formed around a test piece being adhered to the freeze plate had a substantial effect on the shear stress. In other words, the ice worked as a good support material. This effect was quantified by testing with and without this support layer.

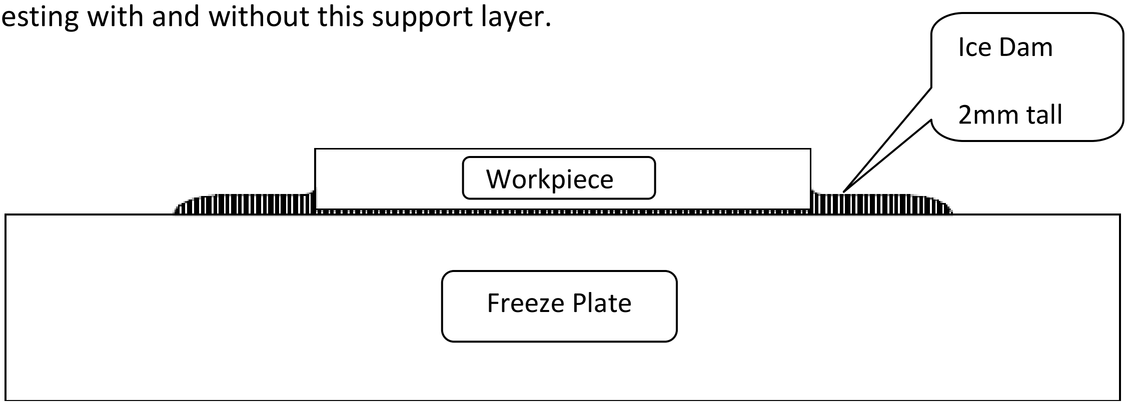

Figure 28 - Ice damming occurs when ice surrounds the sides of the test piece.

By analyzing the layer of ice left on the surface of the freeze plate and the test piece, it is determined that wicking away the excess water on the plate until there is only a small fillet of liquid all around the test piece leaves a full layer of water underneath the test piece. Remembering that the ice is dyed blue, the layer is intact so long as blue can be seen all around the perimeter of the piece. The high surface tension of water tends to equal out the width of the fillet, allowing wicking away of the water until the total width is less than 1mm at the outer edge. As a concave fillet, the actual volume of water left around the part is almost insubstantial.

As with all adhesive joints, cleanliness and fitment have a large effect on adhesion. Pieces that are not properly flat, or which have surface contaminants, can have a bond so weak that it can easily be broken with the fingers. The tendency of aluminum to lose its surface energy and return to a low energy state in a short period of time becomes apparent during testing, and must be accounted for.

6.2 Test Procedure

Before testing, all test pieces were thoroughly cleaned with acetone at least three times to rid them of all surface contamination. Pieces were lapped flat on a granite surface plate using P150 sandpaper. Some of the aluminum pieces were lapped to P400 and P800 to measure the effect of surface roughness on bond strength.

The table which held the test pieces was cleaned three times with acetone and a clean zone was marked off. A new pair of gloves was worn any time hands needed to come in contact with the test surfaces of pieces, though this was rare.

In general, the plate and the pieces were dried off with a fresh paper towel between each test and a new layer of water was used. The freeze plate was returned to equilibrium temperature between every test. A new layer of water was sprayed on the plate and the piece placed firmly, then excess water was wicked from around the piece until there was only a small fillet of no more than 1mm around the piece. Care was taken not to move or disturb the piece during this period.

After the part had completely frozen down, at least 30 seconds after the plate had reached minimum temperature, a bolt was passed through the test piece or a steel cage placed around it and attached to the cylinder via a chain. Pressure was added to the system at no more than 1 psi per second until the chain tensed, after which the chain was checked for alignment to ensure a straight pull. Pressure continued to be added until the piece broke free, and the gauge was read five seconds later when equilibrium had been reached. Examples of a test setup before and after shear are shown in Figure 29 and Figure 30.

Pieces were examined to determine what percentage of the ice under the piece had broken off the plate. In cases where there is little or no ice left on the piece, such as in Figure 31, it can be safely assumed that the adhesive force of the piece had been accurately measured. If the majority of the ice stays on the piece, then the plate side of the adhesive joint has failed and the measured shear force can only be a lower bound as in Figure 32.

Parameters noted in the test results include pretreatment of the piece and the freeze plate. These treatments include wiping dry, torching, and cleaning with acetone and the order in which they were performed. Treatments other than cleaning were only performed on the plate as they could substantially skew the results, and the most useful tests are those where the bond fails completely at the test piece side. For all tests, the plate was cooled from a starting temperature of roughly 15 degrees Celsius to a final temperature of -12 degrees.

The aluminum test pieces were made up several days in advance of the first test to prevent their recent abrasion from having an effect on the results. The pieces measured 1x1x1” and 2x1x1” and have three of their four sides lapped to P150, P400, and P800 grit on the granite surface plate. Other test pieces were prepared at the time of testing, and include a shell test piece, a steel test piece, a wood (ebony) test piece, and an acrylic test piece all lapped with P150 grit.

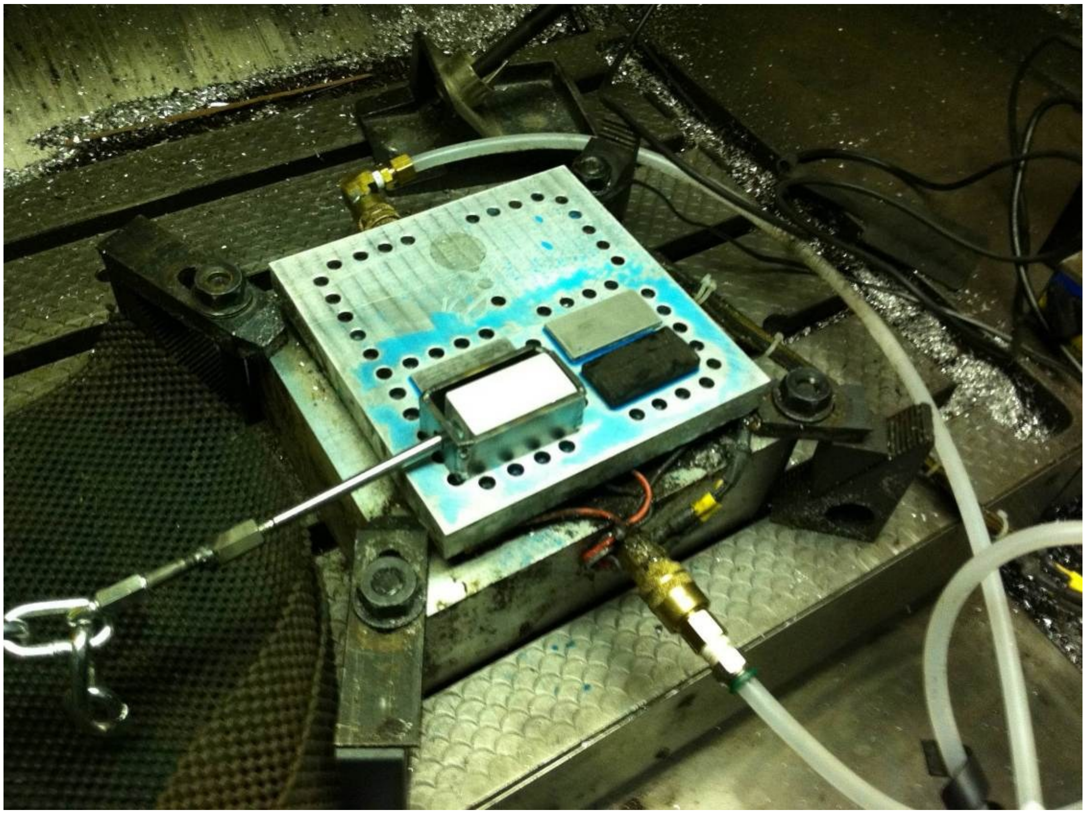

Figure 29 - Full plate of workpieces, steel pulling cage shown around aluminum

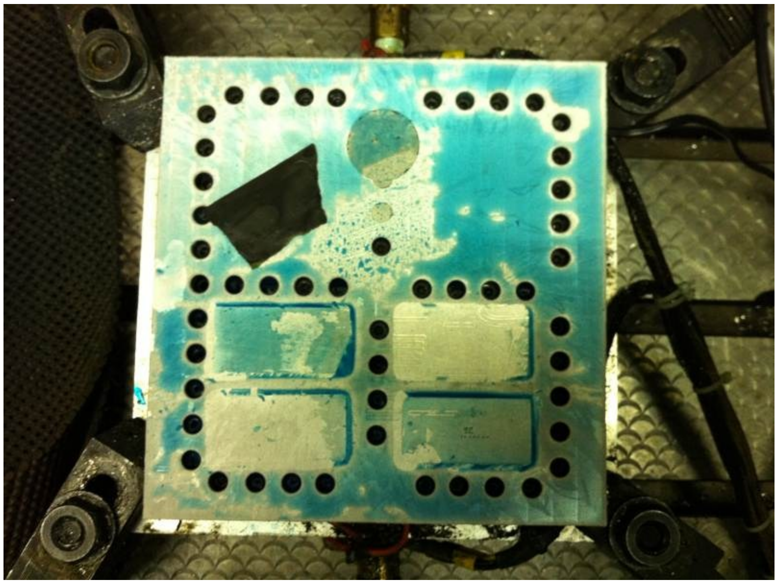

Figure 30 - Same plate after tests

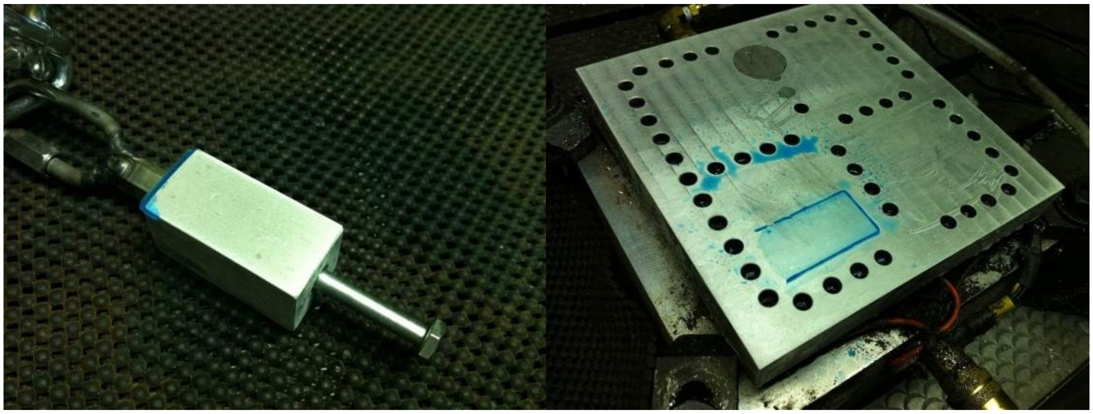

Figure 31 - Test piece and plate showing typical test piece side failure

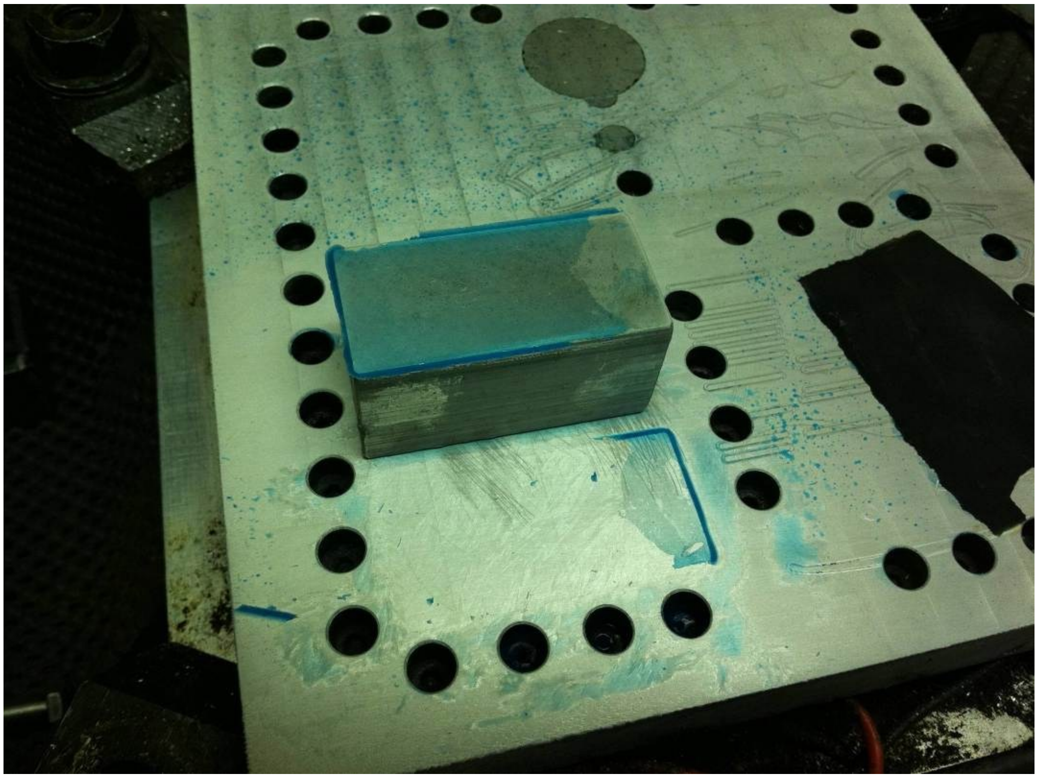

Figure 32 - Typical plate side adhesive failure

The aluminum test pieces, the freeze plate, and the shell test piece were measured for surface roughness with five tests each at various positions on the sample. Tests were all done in the direction the pieces were to be pulled during shear tests. Results are shown in Figure 33.

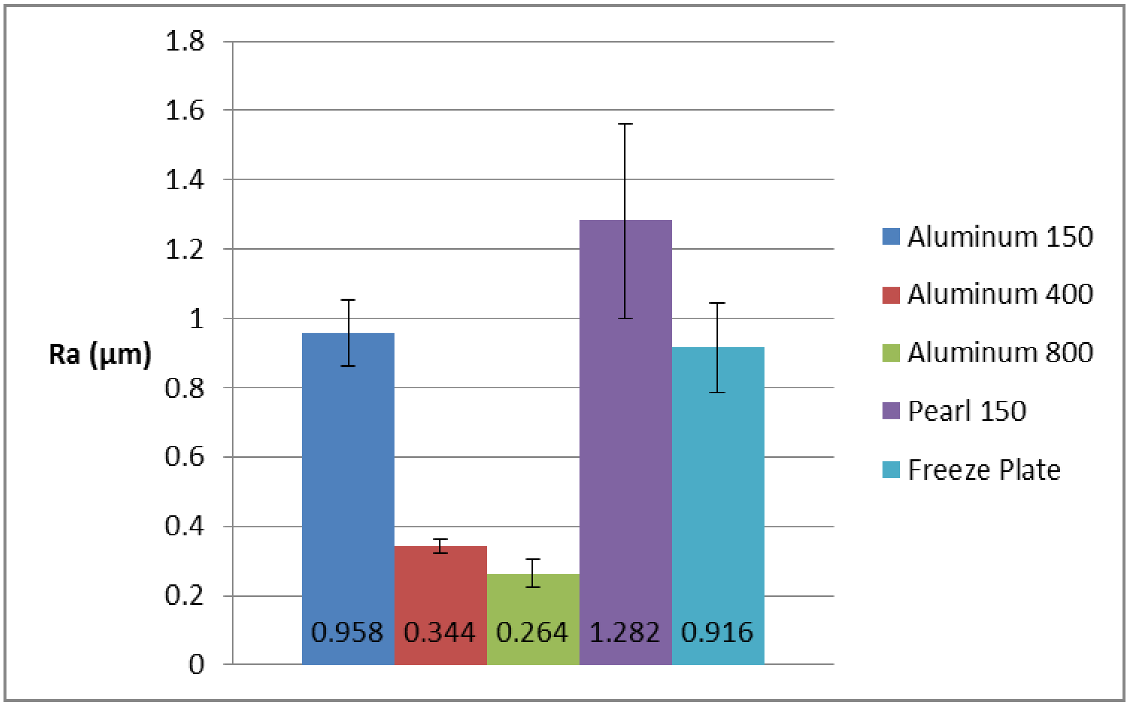

Figure 33 - Test piece and freeze plate surface roughness

6.3 Test Results and Analysis

The initial test was done with a 1" aluminum cube to act as a baseline for the rest of the results. This is when the ice damming effect was first noticed. The dammed results were laid in a pool of water which was not altered in any way before freezing, and thus had a 2mm dam of ice all around them. As can be seen in Figure 34, this has a substantial effect on the shear force versus the pieces which had all water around the aluminum removed. The number of test pieces is noted inside the columns of the chart. It is also interesting that the dammed pieces tend to hold onto a higher percentage of the ice when they separate, hypothetically due to the continuity of the ice surface all the way around and underneath the piece.

The average of the undammed values, 762 kPa, is roughly equal to the values found in (F Hassan et al., 2010) at 782 kPa for 80 grit roughness. These are much lower than the results in (Haehnel, 2002), averaging 1500 kPa, and it is suspected that this is due to the possibility of mixed mode failure caused by the small surface area and high loading point of the test block.

It is clear from the test results that ice damming can add a substantial amount of holding force. Though it is avoided through the rest of the testing to keep results consistent, in a real world situation it would be a beneficial effect. The 450 kPa added by ice damming could double the effectve shear strength of bonds with low energy materials such as shell and acrylic. Ice damming is a measure of ice as a potting compound, and it could be valuable in future research to quantify its effects over a larger sample of materials and surface areas as a practical reference for using this technology.

With a baseline established, tests were continued from that point only with 2” by 1” samples, shown in Figure 35. Using larger samples averages out small errors due to surface irregularities and allows the samples to seat themselves in the water pool simply due to mass. The same procedure of wicking the water from underneath the sample was used. The pieces are pulled on their long axis, which should minimize the effect of torsional forces on the results.

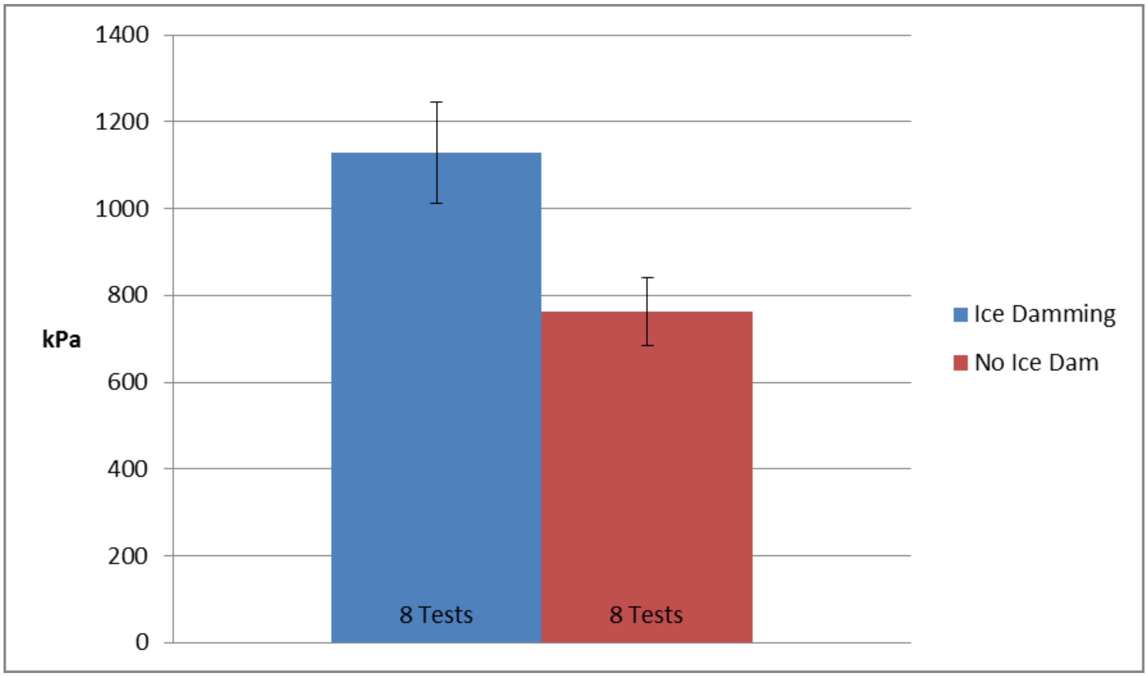

Figure 34 - Comparison of shear with and without ice damming

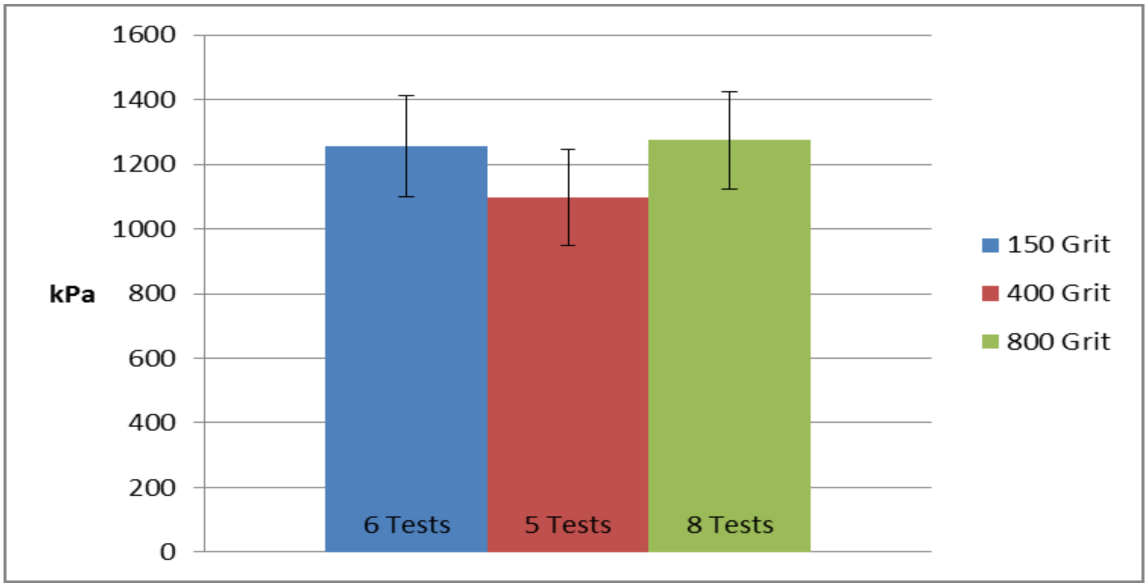

Figure 35 - Shear of aluminum pieces, by grit

The tests in Figure 35 show that a high and consistent bonding strength doesn't necessarily depend on surface roughness. This is a distinctly different result than those in papers correlating roughness to bond strength. (F Hassan et al., 2010) found a sharp decrease in holding strength, roughly 75%, with decreasing surface roughness on pieces sanded from 80 to 400 grit, with aluminum lapped to 400 grit shearing at only 190 kPa. (Zou et al., 2011) found that the holding strength of as received aluminum samples, very close to the roughness of an 800 grit sample, had a maximum of 330 kPa. This may speak further to the role of fitment in the adhesive ability of ice, or possibly to increased shear strength when in a thin layer. In tests done by Zou et al. and Haasan et al., the ice was bonded to only one surface and broken off to test bond strength. In tests by (Haehnel, 2002), with shear strengths similar to the current results, ice was also tested in shear between two surfaces.

Some of the results from these tests are difficult to analyze because a large number of samples broke off with a full ice surface, meaning they exceeded the bond of the freeze plate to the ice. Surfaces from P150 to P800 can all achieve shear strengths exceeding 1400 kPa. Given that both the current research and (Haehnel, 2002) controlled more strongly for direction of force application as well as fitment, the author hypothesizes that the lower shear strengths measured in other testing were the result of other failure modes such as torsion or peeling which had not been fully accounted for in the test setup.

Dividing the results by surface treatment done to the plate, Figure 36 makes it clear that treatment can have a substantial effect on adhesion. It is interesting that this effect is highly apparent given that only the plate was treated with the torch, never the test pieces. Regardless, the data shows that torching is substantially more effective than cleaning alone. Torching twice, with a thorough cleaning in between, is shown to be superior to all other methods and was used in all of the strongest bond results. Given the relatively low expense of propane, it can only be recommended that torching be used in all cases where there is any doubt of sufficient adhesive force in this type of clamping. Speculation on how torching only one surface can cause a better bond to both surfaces leads to a hypothesis that the full wetting of the plate prevents water from being pushed away from the test piece as easily. As water is more difficult to remove from the interface, it would make sense that voids and areas of surface that might have avoided contact before would become wetted with greater contact time. Regardless of its mechanism of action, flame treatment is certainly effective and an important discovery given its absence from current literature on ice adhesion.

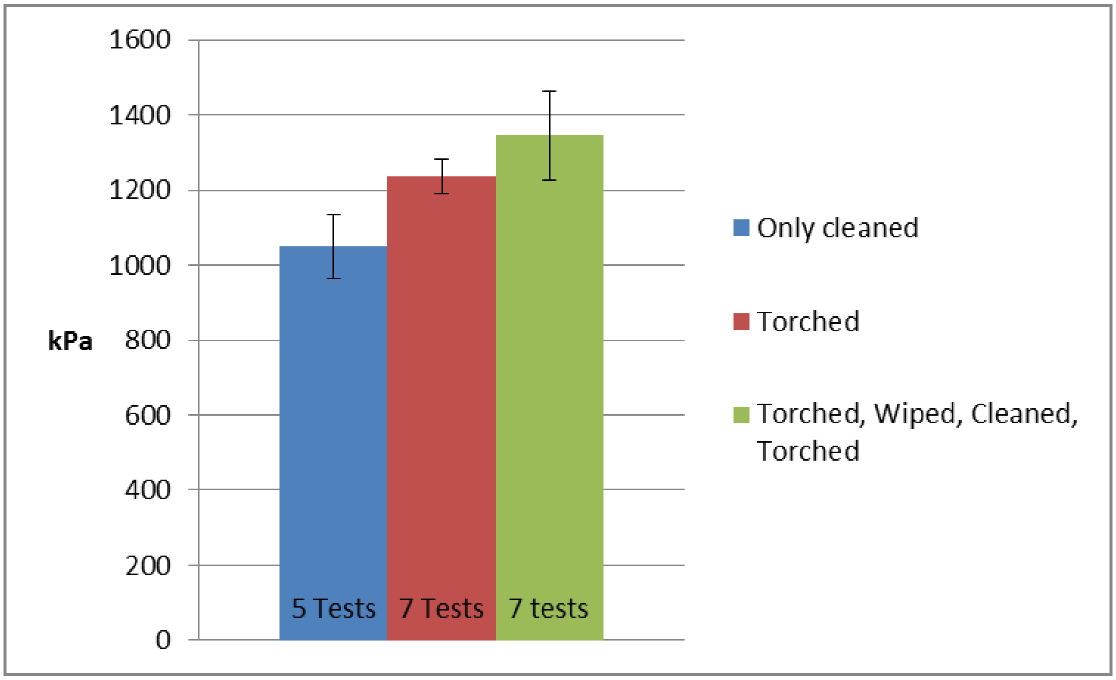

Figure 36 - Shear strength by surface preparation of plate

To simulate real world conditions, a small set of tests was done with a variety of materials using all four positions on the freeze plate with their results shown in Figure 37. Earlier tests were done using only one position for consistency, but for relative values which can be used to make a practical decision it was unnecessary and possibly more realistic to use less controlled conditions. The pieces were placed and replaced on the plate during subsequent tests, with no preparation done to the pieces or the plate besides drying with a paper towel between tests. It is believed that this protocol shows the minimum results which can be expected in real world production situations barring gross operator error. While the minimum achieved shear values are only one third of those for strong magnetic chucks, they are still more than five times the forces expected from vacuum clamping. That is more than sufficient for the sort of light machining done on the delicate parts the process is most suited for.

The result for steel is similar to that seen in (Meuler et al., 2010), and the aluminum value is similar to the less controlled tests done initially in the current research. Ebony shows good wetting, but holding force decreases on subsequent tests as the piece tends to warp as water is taken up. In working with wood, it is suggested that pieces only be attached once or are dried between operations, and that a minimal layer of water is used. Both shell and acrylic wet very poorly, but their adhesion values are quite acceptable given that small parts in either material tend to be quite intricate and thus have low machining forces. Given that shell has been shown to be machinable using this process, the other materials appear to have holding power in reserve.

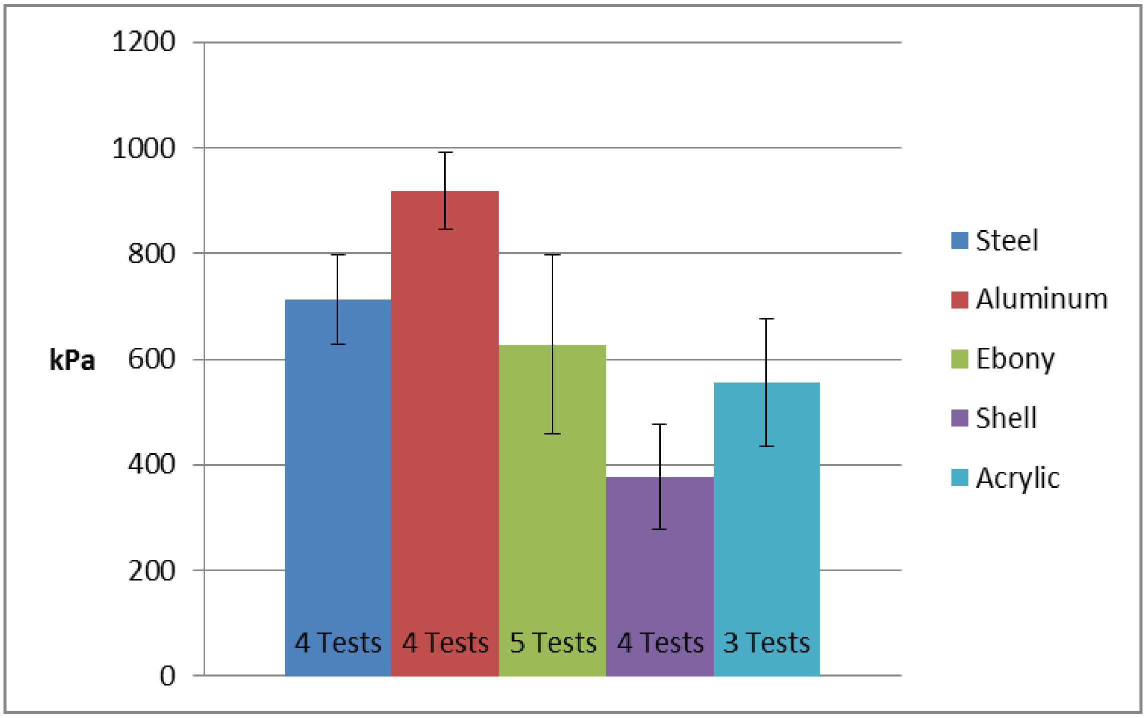

Figure 37 - Comparison of material bond strength, no part or plate prep besides drying

As the mode of bond failure was tracked throughout the tests, it makes sense to analyze it in terms of bond strength. The specific variable tracked is the percentage of ice attached to the test piece after failure, with 100% being a complete failure on the plate side and 0% being a full failure on the test piece. A result of 0% gives an absolute value for test piece adhesion, whereas a result of 100% gives only an upper bound on the adhesion of the ice to the test piece. It can be seen from Figure 38 that values near zero and 100 correspond to low and high shear strengths, with mixed mode failure on both test piece and freeze plate occurring at a wider range of shear strengths.

The R^2 value of this plot is shows good correlation, and so given no other information it can be assumed that analysis of the ice attached to a workpiece can provide a basic idea of adhesive strength to a particular freeze plate. The most important real world application of this finding is in post failure analysis. In a production environment, the shear stress of a bond failure will not be measured and so the use of dyed water as an aid in troubleshooting failures seems well advised.

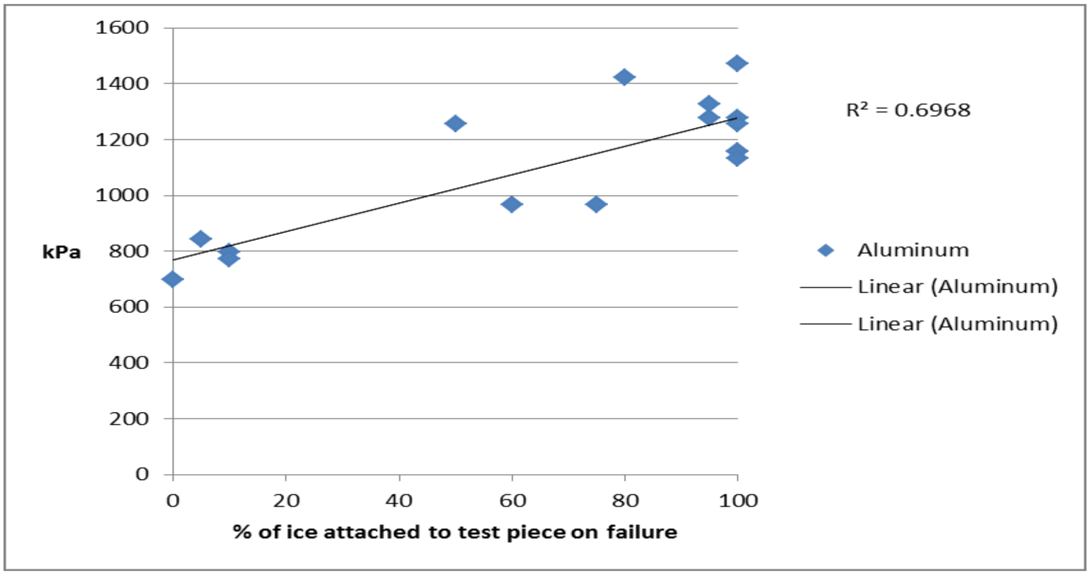

Figure 38 - Comparison of bond strength with bond failure surface

As the purpose of the device is to aid in machining of small components, cutting test pieces were run using both shell and aluminum. Images of cutting are shown in Figure 39 and Figure 41. The shell piece was chosen to test the setup on delicate workpieces, and the aluminum was chosen to test harsh machining conditions.

The shell piece shown in Figure 40 has a line width less than 0.015” with a piece depth of 0.06”. Shell is very difficult to hold using most adhesives; only hide glue and CA are effective. In general, a piece longer than one inch at that line width would be nearly impossible to release from hide or CA glue and clean without damaging it; yield can be ½ per inch of line length on designs below 0.015”. Figure 40, at 5” of linear length, would have required up to 32 attempts to yield a completely perfect result. Increases in yield can be had by decreasing feed rate or depth of cut correspondingly, but the cost of increasing cut time is even larger than the material cost for multiple attempts. Using ice to fixture the shell, a number of consecutive successful pieces were cut.

The aluminum test piece is a large block, meant to test relatively harsh maximum operating parameters. A ½” end mill was used to face the block at 200 inches per minute feed rate, 10 000 RPM, and a 0.1” depth of cut with no detachment. Though aluminum normally requires coolant to cut at high speed without galling, the temperature and thermal mass of the block prevented this behavior even while cutting dry. Those conditions are far beyond the cutting forces which would be placed on the type of complex components that this device would be used for, but overhead capacity at no cost is never a bad thing.

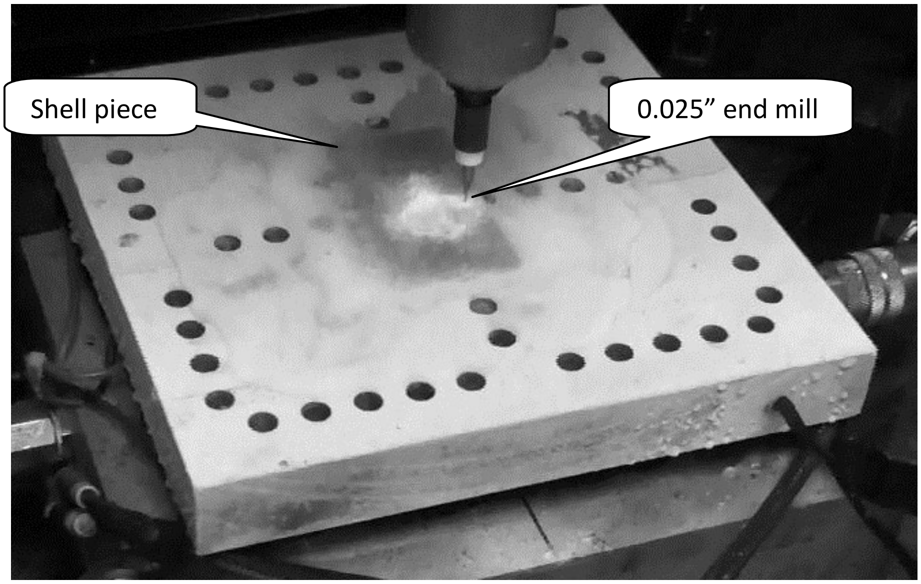

Figure 39 - Cutting shell test piece on the freeze plate. Piece on plate (covered in dust) is shown in Figure 40. Cold plate is 8” square.

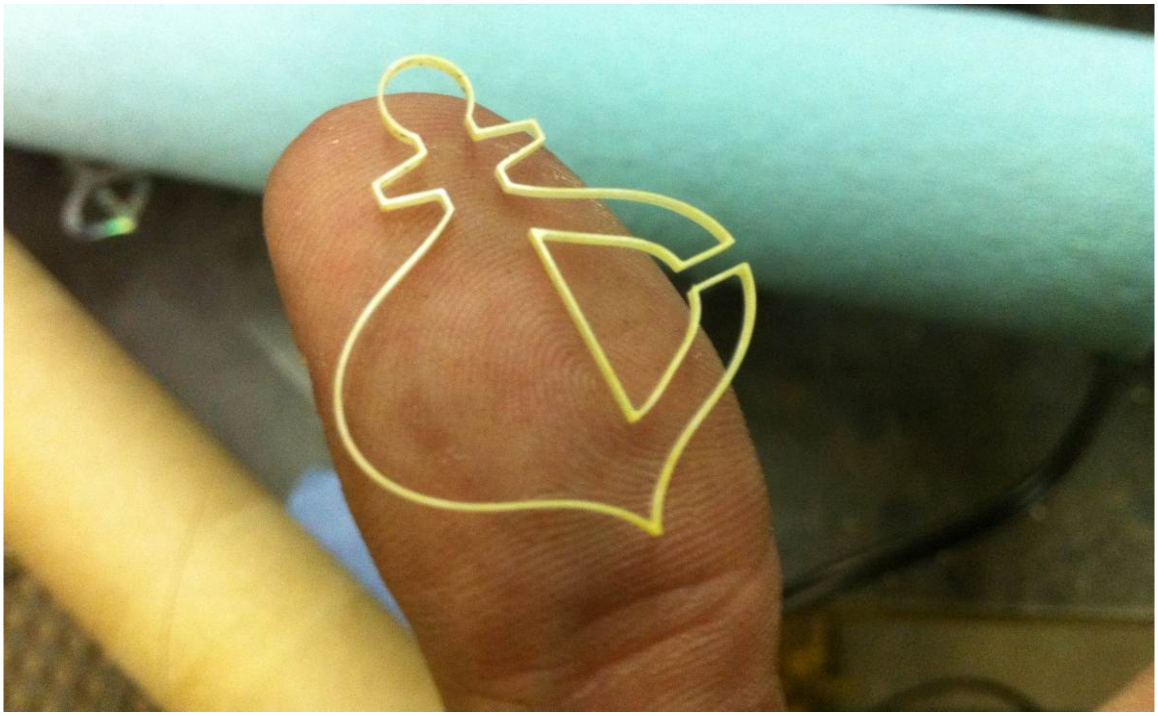

Figure 40 - Shell test piece. The piece fits inside a 28mm circle (standard Canadian Toonie), and line width is 0.014”.

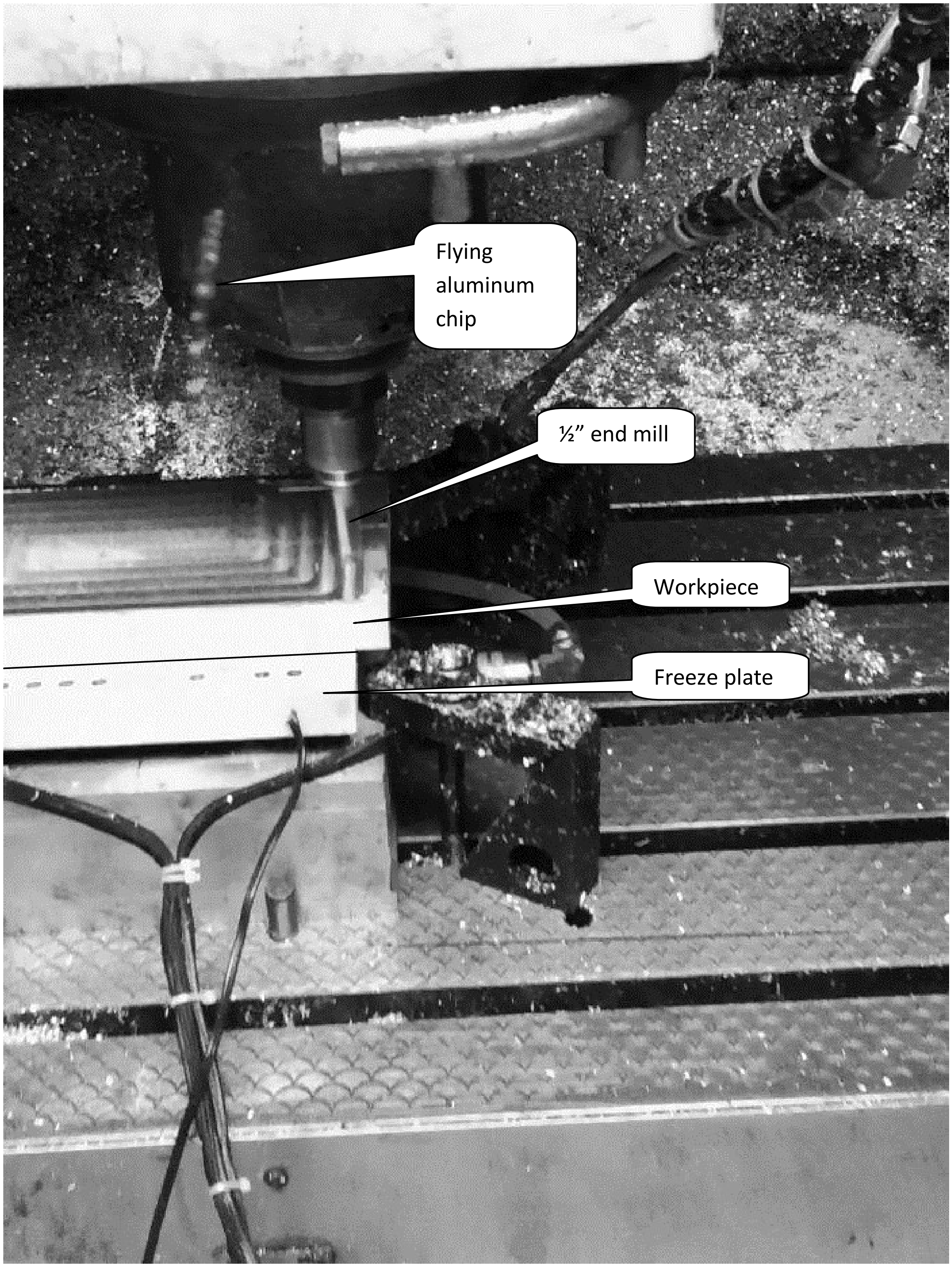

Figure 41 - Milling aluminum, blur at upper left is a chip in midair. No galling occurred despite lack of coolant usage.

Chapter 7 - Conclusion

Specific Contributions:

Contributions to the academic community from this research include:

- Design and implementation of a low cost freeze chucking system for research, with freezing speed matching or exceeding commercial devices.
- Development of a simple and inexpensive test method and apparatus for measuring the performance of such devices.
- Cutting tests proving that this method is viable for manufacturing extremely small and delicate parts, currently uneconomical or impossible otherwise
- Showed utility of optimizing ice adhesion strength through surface preparation, which resulted in attaining very high adhesive bond strengths.
- Examinations of 'ice damming' and flame treatment on shear strength of ice bonds with other materials.
- Measurements of ice adhesion strength in production conditions (minimal surface preparation and cleaning) on a variety of materials commonly used for fragile components.

The current research shows that fixturing with ice is a viable method for workholding, particularly for small and delicate parts, and also that the holding forces can be quite large. If not particularly useful in terms of the work done, having holding force in reserve is certainly comforting insurance when machining delicate and highly expensive components. The research took a turn in a different direction than planned when the large variance in holding forces due to part and surface preparation was noticed in early experiments. It is believed that this data is of even more merit than the originally planned survey of freezing temperatures and materials as it is paramount for success in holding any material with ice, whether it is a high energy material which might be easily held in the first place or a low energy material which is difficult in all cases. The initial tests on acrylic had such poor holding force that the parts could be removed by hand, and even the initial shear tests on aluminum had holding forces of only 20 psi.

The major finding, and contribution, of this research is the large effect that ice damming and flame treatment can have on shear strength in bonds with ice. Though prior research characterized the basic properties of these bonds quite well in many cases, there had not been a focus on increasing bond strength for use in adhesive of fixturing applications. In fact, nearly all other research in the area is focused on decreasing the adhesive strength of ice and thus doesn't consider factors which could increase bonding strength at all.

The cutting tests on shell, an extremely low surface energy material, and using large forces on aluminum show that this method is viable for real world machining problems and not simply an academic curiosity or super niche process. Besides providing superior results to any other method for cutting detailed pieces in shell, up to a 32 fold increase in yield, the cutting tests in aluminum show that the method is a viable way of holding general metal cutting workpieces. The utility of this might not be immediately apparent until one considers that often in medical and high tech fields delicate parts must be worked on in cleanroom level environments where both coolants and high pressure fixtures are simply not an option. In shell, which has machining issues similar to graphite and ceramic, this method has made forms possible which were not previously so except through use of prohibitively expensive potting methods using soluble polymers.

Work which would be of value in this area but which was beyond the scope of these experiments could certainly include explorations into the degradation of the ice bond due to heat or vibration inputs. As a brittle material, ice may be particularly susceptible to vibrations which are accepted as normal operating procedure in machining.

Ice damming could be quantified further with more research, which could be an important contribution to machining practice using this technology. Torch treatment of workpieces was avoided in this research in order to obtain results which could be directly compared to the literature results, but it would certainly be of benefit to look into the possible applications to both low and high energy workpiece materials.

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