Ceramic-aluminum bonded joints for high temperature applications

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Abstract

This project is about ceramic-aluminum bonded joints for high temperature applications. Most of the current solutions used in high performance engineering applications are limited to a maximum of 260ºC (RTV). Combination of adhesives has been previously demonstrated as a possible method to improve the behavior of the joint in high temperature loadings. Ceramic adhesives, high temperature adhesives and dual adhesive are all considered. The experimental work presented in this study consisted in measuring the temperature distribution bond line for a sandwich composed of aluminum-adhesive-ceramic. The temperature was measure with thermocouple worded in the bond line and an infrared camera directed to the ceramic tile. The result shows that the device developed grins a uniform temperature in the adhesive and simulate properly the ceramic shields of aerospace vehicles.

ACKNOWLEDGEMENTS

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1. Introduction in Adhesive and Sealants Bonding

This introduction gives basic notions about adhesive and sealant bonding. Adhesives and sealants were first used many thousands years ago. The development of modern polymeric adhesives and sealants began about the same time as the polymer industry itself, early in the 1900s.

With successful experiences in these industries, it was soon realized that adhesives could be used to economically replace mechanical fastening methods such as welding, brazing, or riveting.

Adhesives and sealants are used in a variety of industries: construction, packaging, furniture, automotive, appliance, textile, aircraft, and many others. A large number of manufacturers supply many different products to numerous end-users for a multitude of applications.

The industries that are most influenced by adhesives and sealants consist of four main categories:

- Base material producers including resins, mineral fillers, extenders, etc.
- Formulators who take the base materials and combine, process, and package them into adhesive and sealant systems that provide various levels of performance.
- End-users who take the packaged adhesives and sealants and produce assembled products.
- Associated industries such as equipment manufacturers, testing laboratories, consultants, etc.

Adhesive and sealant bonding involve some advantages and disadvantages compared with another method, and these are:

Advantages and Disadvantages of Adhesive Bonding [1]

Advantages	Disadvantages
1. Provides large stress-bearing area.	1. Surfaces must be carefully cleaned.
2. Provides excellent fatigue strength.	2. Long cure times may be needed.
3. Damps vibration and absorbs shock.	3. Limitation on upper continuous operating temperature.
4. Minimizes or prevents galvanic corrosion between dissimilar metals.	4. Heat and pressure may be required.
5. Joins all shapes and thicknesses.	5. Jigs and fixtures may be needed.
6. Provides smooth contours.	6. Rigid process control usually necessary.
7. Seal joints.	7. Inspection of finished joint difficult.
8. Joins any combination of similar or dissimilar materials.	8. Useful life depends on environment.
9. Often less expensive and faster than mechanical fastening.	9. Environmental, health, and safety considerations are necessary.
10. Heat, if required, is too low to affect metal parts.	10. Special training sometimes required.
11. Provides attractive strength-to weight ratio.	

The major disadvantage are :

- The storage life of the adhesive may be unrealistically short; some adhesives require refrigerated storage.
- The adhesive may begin to solidify before the worker is ready.
- The cost of surface preparation and primers, if necessary, must be considered.
- Ease of handling, waste, and reproducibility can be essential cost factors.
- Cleanup is a cost factor, especially where misapplied adhesive may ruin the appearance of a product.
- Once bonded, samples cannot easily be disassembled; if misalignment occurs and the adhesive cures, usually the part must be scrapped.

Many of these hidden costs can be minimized by the proper choice of adhesives and processes.

General requirements for all adhesives and sealants [1]

If one looks at the adhesive bonding or sealing ‘‘process’’ as a complete procedure, encompassing all aspects of material selection, joint design, production, etc., then the basic requirements are the same no matter what the application. These universal requirements for successful application are:

1. Cleanliness of the substrate surface
2. Wetting of the substrate surface (intimate contact of the adhesive or sealant on the substrate)
3. Solidification of the adhesive or sealant
4. Forming a ‘‘joint’’ structure (adhesive or sealant material, interphone regions, and adherents) that is resistant to the operating stress and environment
5. Design of the joint
6. Selection and control of materials and manufacturing processes.

2. Adhesive Classifications

Synthetic adhesives are manufactured from man-made materials such as polymers. Natural adhesives are manufactured from naturally occurring materials such as animal or agricultural by-products. The industry has settled on several common methods of classifying adhesives that satisfy most purposes.

These classifications are by:

- Function
- Chemical composition
- Mode of application or reaction
- Physical form
- Cost
- End-use

2.1 Function

Defines adhesives as being either structural or nonstructural. Structural adhesives are materials of high strength and permanence. Structural adhesives are generally presumed to survive the life of the application. Conversely, nonstructural adhesives are not required to support substantial loads, but they merely hold lightweight materials in place.

2.2 Chemical composition

The classification of adhesives by chemical composition describes adhesives in the broadest sense as being either thermosetting, thermoplastic, elastomeric, or alloys (hybrids) of these.

- **Thermosetting adhesives.** Thermosetting adhesives are materials that cannot be heated and softened repeatedly after their initial cure.

- **Thermoplastic adhesives.** Thermoplastic adhesives differ from thermosets in that they do not cure or set under heat. Thermoplastics are originally solid polymers merely soften or melt when heated.
- **Elastomeric adhesives.** Elastomeric adhesives are based on synthetic or naturally occurring elastomeric polymers having great toughness and elongation.
- **Hybrid adhesives.** Adhesive hybrids are made by combining thermosetting, thermoplastic, or elastomeric resins into a single adhesive formulation.[1]

2.3 Method of reaction

There are several methods by which adhesives can solidify:

- By chemical reaction
- By loss of solvent a mate
- By loss of water
- By cooling from a melt

Adhesives that harden by chemical reaction:

- Two part systems
- Single part, cured via catalyst or hardener
- Moisture curing adhesives
- Radiation (light, UV, electron beam, etc.) curing adhesives
- Adhesives catalyzed by the substrate
- Adhesives in solid form (tape, film, powder, etc.)

Adhesives that harden by solvent or water loss:

a. Contact adhesives
b. Pressure sensitive adhesives
c. Reactivatable adhesives

d. Resinous adhesives

Adhesives that harden by cooling from the melt:

- Hot melt adhesives
- Hot melt applied pressure sensitive and thermosetting adhesives

2.4 Physical form

Adhesive systems are available in a number of forms.

The most common forms are:

- Multiple part solvent less (liquid or paste)
- One part solvent less (liquid or paste)
- One part solution (liquid)
- Solid (powder, tape, film, etc.)

Various Types of Adhesives by Form

Solid adhesive

- Film adhesives (unsupported)
- Tape adhesives (supported)
- Solid powders and preforms
- Solvent based adhesives and primers
- Hot melt adhesives

Liquid adhesives (100% solids paste and liquid)

a. One-component: long shelf life

- heat cured
- cured by surface or anaerobic catalysts
- cured by exposure to ultraviolet (UV) light, radiation, or some other energy source

b. Two-component: short pot life

- room temperature cured
- heat cured

Liquid solvent containing adhesives (similar to 100% solids systems above but with solvent for viscosity reduction)

a. Solvent based contact adhesives

b. Water based adhesives

- contact or pressure sensitive adhesives
- solutions or emulsions

c. Pressure sensitive adhesives

Cost is usually not used as a method of classifying adhesives; however, it is an important factor in the selection of a specific adhesive and a factor in determining whether adhesives should be used at all.

The following parameters may be important in analyzing the real cost of an adhesive system:

- Efficiency of coverage in relation to bonding area or number of components
- Ease of application and processing equipment needed (jigs, ovens, presses, applicators, etc.)
- Total processing time (for assembly, for preparation of adherends, for drying, for curing, etc.)
- Cost of labor for assembly and inspection of the bonded parts
- Waste of adhesive contributes to material costs and environmental costs for disposal
- Amount of rejected material as compared with other methods of joining

Depending on viscosity, liquid adhesives can be considered to be sprayable, brushable, or trowelable.

2.5 Materials used for adhesives and sealants

Adhesives and sealants are highly formulated materials with many components. They generally have an organic base, although there are some mineral-based adhesives that perform very well in certain applications.

The modern organic materials are all superior polymers: long chains of carbon atoms that can form three-dimensional networks. The oldest adhesives and sealants, many that are still used today, are of natural origin. The most common of these are animal glues, starches, and tar or pitch. Naturally occurring adhesives and sealants also include dextrin, asphalt, vegetable proteins, natural rubber, and shellac.

For a material to be a potential adhesive or sealant, it must meet three basic criteria. First, the material has, at some stage, to be in a liquid form so that it can readily spread over and makes intimate contact with the substrate. Second, the material must be capable of hardening into a solid to withstand and distribute stress. Lastly, the material must resist the environments that it will see during processing and during service.

Modern polymers are ideal materials to use as adhesives and sealants because they can be applied as low viscosity liquids and, then by various means, hardened into a strong material with relatively good resistance to stress and various environments. [1]

Classification by origin, basic type, and chemical family of common adhesives will be included because they have wide use in certain applications, and this classification symbol in the table 1.

Table 1. Classification by origin, basic type, and chemical family of common adhesives [1]

Origin	Basic type	Family	Examples
Natural	Animal	Albumin/Animal glue/Casein/Shellac/Bees wax	
	Vegetable	Natural resins /Oils and waxes /Proteins Carbohydrates	Gum arabic,tragacanth, colophony, Canada balsam/ Carnauba wax, linseedoil/Soybean/Starch/dextrins
	Mineral	Inorganic minerals/Mineral waxes/Mineral resins/Bitumen	Silicates, magnesia, phosphates, litharge, sulfur/Paraffin/Amber Asphalt
Synthetic	Elastomers	Natural rubber Synthetic rubber Reclaimed rubber	Natural rubber and derivatives Butyl, polyisobutylene, polybutadiene blends, polyisoprenes, polychloroprene, polyurethane,silicone, polysulfide,

			polyolefins
	Thermoplastic	Cellulose derivatives Vinyl polymers and copolymers Polyesters (saturated) Polyacrylates Polyethers Polysulfones	Acetate, acetate-butyrate, caprate, nitrate, methyl cellulose, hydroxyl ethyl cellulose, ethyl cellulose, carboxy methyl cellulose Polyvinyl acetate, polyvinyl alcohol, polyvinyl chloride, polyvinylidene chloride Polystyrene, polyamides Polyacrylates Methacrylate and acrylate/polymers, cyanoacrylates Polyethers Polyhydroxy ether, polyphenolic ethers
	Thermosetting	Amino plastics Epoxies Phenolic resins and modification Polyesters (unsaturated) Polyaromatics Furanes	Urea and melamine formaldehydes Epoxy polyamide, epoxy bitumen, epoxy polysulfide, epoxy nylon Phenol and resorcinol formaldehydes, phenolic-nitrile,

			phenolic-neoprene, phenolic-epoxy Polyimide, polybenzimidazole, polyphenylene Phenol furfural

2.6 Selection of Adhesives

The adhesive selection will be dependent primarily on:

- The type and nature of substrates to be bonded
- The methods of curing that are available and practical
- The expected environments and stresses that the joint will see in service

2.7 Planning for the Bonding Process

The following approach is often recommended when using engineering adhesives.

A) Consider alternative assembly and bonding processes.

B) Gather all information possible regarding the resulting product and its requirements.

C) Design the joint especially for the method of bonding that is chosen.

Joint designs should consider:

- The type of substrate
- The cost associated with manufacturing the joint design
- Ease at which the joint can be fabricated and assembled
- The ease in which the joint can be inspected after bonding is complete
- Most importantly, the type of stress that the joint will see in service

D) Choose the appropriate adhesive in cooperation with an adhesive supplier. Estimate the required adhesive strength and durability.

F) Ensure the reliability of the adhesive bond by engaging in a testing program to verify the durability of the joint by simulating actual assembly practices and aging environments.

2.8 Selecting the Adhesive

The best adhesive for a particular application will depend on the materials to be bonded, the service and assembly requirements, and the economics of the bonding operation.

2.9 Substrates

Generally, the first consideration in making an adhesive selection is the type of substrates that must be held together.The type and nature of the materials to be bonded and the surface preparation that they may require are prime factors in determining which adhesive to use. Some adherends such as aluminum or wood can be successfully bonded with many adhesive types; other adherends such as nylon can be bonded with only a few. The substrate type, condition, porosity, finish, acidity, alkalinity, etc. will all influence the adhesive selection. These stresses could result in premature failure of the bonded components. Incompatible adhesive and adherend can also lead to damage of an assembly.

- **Adhesives** for bonding metals may be divided into two main groups:
  - Structural—for use in continuously stressed structures
  - Non-structural—for use in low strength applications
- **Plastics**

Bonding of plastics is generally more difficult than metal bonding because of the lower surface energy of polymeric substrates.

Elastomers

- Vulcanized elastomers.
- Unvulcanized elastomers.

Other common substrates: wood and glass [1]

2.10 Polyimide Adhesives

Ever since polymeric materials were introduced to join metal adherends in environmentally harsh structural applications, there has been an ever-growing demand on synthetic chemists to develop newer, more durable, high performance adhesive systems to meet the above challenges. Aromatic polyimides are among the most studied family of adhesives for applications requiring resistance to high temperature or aggressive environments [2]. This group LITERATURE REVIEW 20 of materials was first synthesized commercially in the 1960s and have since found a wide range of applications [2]. Some advantageous features of polyimides that make them very attractive for aircraft applications are thermo-oxidative stability, radiation and solvent resistance, and excellent mechanical performance.

Polyimides are generally divided into two groups, thermoplastics and thermosets.

Thermoplastic polyimides are mostly formed by a step-growth condensation reaction, between an aromatic diamine and an aromatic dianhydride. This reaction may be a one-step or a two-step process, with the intermediate formation of the polyamic acid in the two step process [2].

Unlike thermoplastic polyimides, thermosetting polyimides experience a chemical transformation during cure that renders the material thermally nonprocessible. The crosslinking reaction that occurs in the formation of thermosetting polyimides is often a chain growth mechanism, or addition polymerization between an aromatic diamine and an aromatic dianhydride. These polyimides offer certain advantages over thermoplastic polyimides in that they possess better solvent resistance, higher glass transition temperatures, and less creep [2].

2.11 Ceramics adhesive

They in present do not offer a hermetic connection or high degrees of reliability. Their reliability and resistance is below that offered by high performance ceramics, and this can result in cracking under thermal stresses. The use of these adhesives is therefore a very complex proposition and requires careful design. A joint bonded with ceramic adhesives can be inspected and tested using scanning electron microscopy, mechanical testing and thermal imaging. The use of two adhesives with different thermal behavior combined can result in joints with exceptional properties.

Experimental work performed with polymeric adhesives also demonstrates that the resistance of the joint at high temperatures is somewhat inferior to the resistance of the high temperature adhesive. A similar effect is seen at the lowest temperatures.

Ceramic materials have been combined with high temperature organic adhesives to provide additional temperature resistance. Tests done with formaldehyde resin and ceramic fillers have identified some of the factors responsible for this effect.

Metal-ceramic connections show a combination of different chemical bonding types: some of the bond regions exhibit pairs of positive and negatively charged particles along the interface, creating ionic connections; metallic/covalent connections make up the rest of the connections observed along the interface/ Connections between metal and ceramic therefore always require a good surface preparation, particularly the introduction of an oxide layer in the surface, promoting covalent bonding. [3]

2.12 Silicon adhesive

A silicone adhesive falls into the elastomeric category. It is a flexible bond that can be used as a sealant as well as an adhesive. A benefit of using a silicone adhesive is its good thermal properties. This type of bond is resistant to high heat, operating over a range from -50°C-260°C. The fact that it is so compliant makes it insensitive to vibration and thermal changes in the environment.

In terms of its mechanical properties, silicone adhesives have a Modulus of Elasticity (E) of approximately 300psi, a Shear Modulus (G) of approximately 100psi, and a Bulk Modulus of approximately 100,000psi. This shows that the bonds created by silicone are strong and reliable. Silicone bonds are compliant in shear and tensile stiffness, but are stiff in compression. Silicone adhesives are also insensitive to water, and some actually cure faster in the presence of moisture. These adhesives are also electric isolators. Since they are so insensitive to heat, they can be used in hot areas and in the presence of open flame.

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A silicone adhesive structure is similar to quartz, with a backbone of alternating silicon and oxygen atoms and can be modified by attaching organic groups. Nearly all silicone products are derived from silicone fluids, rubbers or resins. Silicone adhesives have resistance to temperature extremes, water repellency, electrical insulation properties, high flexibility and excellent release properties.

3. Joint design

3.1 Forces in adhesive joints

The design of the adhesive or sealant joint will play a significant factor in determining how it will survive outside loads. Four basic types of loading stress are common to adhesive or sealant joints: tensile, shear, cleavage, and peel. (Figure1). The strength of an adhesive joint is determined primarily by the mechanical properties of the adhered and the adhesive, the residual internal stress, the degree of true interfacial contact, and the joint geometry. Each of these factors has a strong influence on joint performance [5]

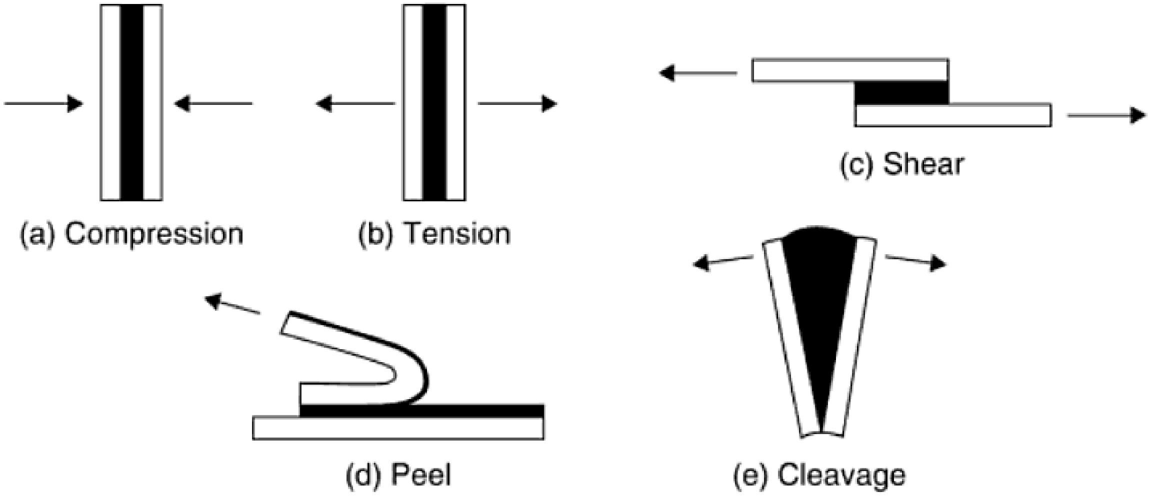

Figure 1. Type of stresses in adhesive joints. [5]

For maximum joint efficiency, non-uniform stress distribution should be reduced though proper joint design and selection of certain design variables that are of importance to stress distribution.

The following variables are most important:

1. Adhesive material properties

2. Adhesive thickness

3. Geometry of the bond area

4. Adherend properties

3.2 Typical joint designs

The typical design of adhesive joints are presented in Figure 2 :

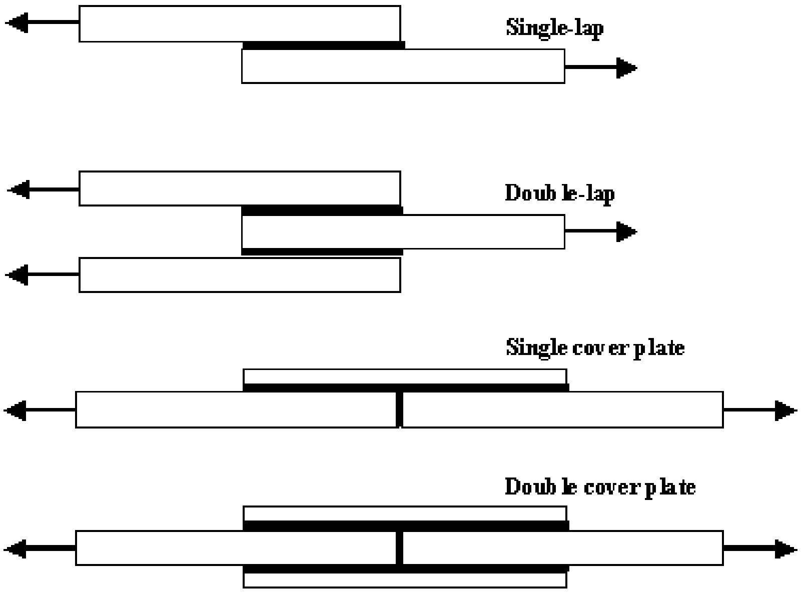

Figure 2 -Typical design of adhesive joints.

a.)Lap joint

These are the most commonly used adhesive joints. They are simple to make, can be used with thin adherents, and stress the adhesive in its strongest direction. The simple lap joint (Figure 3), is offset and the shear forces are not in line. It can be seen in this stress distribution curve that most of the stress is concentrated at the ends of the lap.

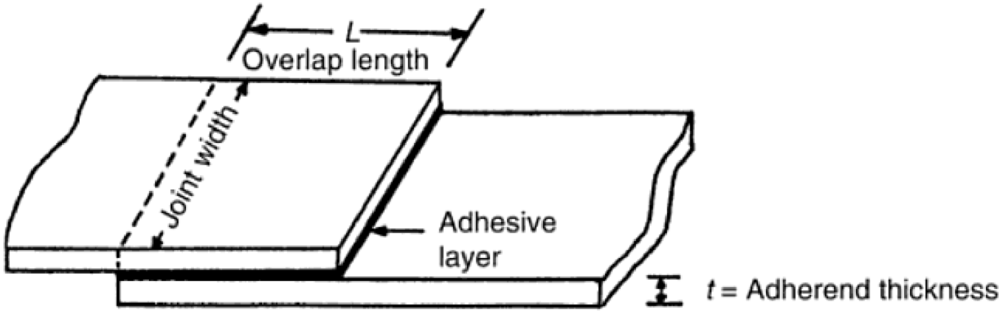

Figure 3 – Single lap joints

Double lap joints. These joints are also more efficient that plain lap joint. The bevelled edges allow conformance of the adherents during loading, with a resultant reduction of cleavage stress at the ends of the joint.

Jaggle lap joints. This is the easiest design for aligning loads. This joint can be made by simply bending the adherents. It also provides a surface to which it is easy to apply pressure.

Strap joints. These joints keep the operating loads aligned and are generally used where overlap joints are impractical because of adherent's thickness. As in the case of the lap joint, the single strap is subject to cleavage stress under bending forces. The double strap joint is superior when bending stresses are involved. The bevelled double strap and recessed double strap are the best joint designs to resist bending forces. However, both these joints require expensive machining. [5]

b.) Butt joints.

These joints are not able to withstand bending forces because under such forces the adhesive would undergo cleavage stress. If the adherents are too thick to design simple overlap joints, modified butt joints can be designed. Such joints reduce the cleavage effect caused by side loading

c.) Strap joints.

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3.3 Stress in a simple lap joints

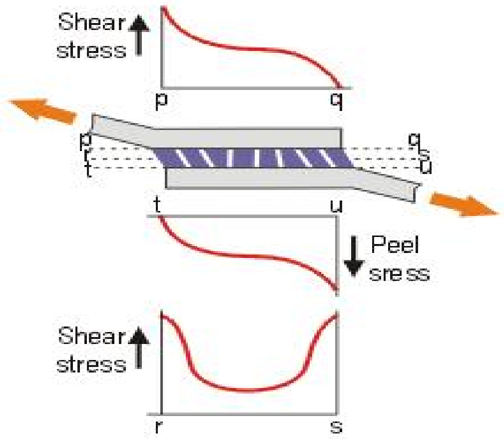

Figure 4 – Distribution of stress in a simple lap joints

Figure 4 show the stress in a simple lap joint made from thin metal sheet :

Shear stress is across the adhesive bond. The bonded materials are being forced to slide over each other.

Peel stress is the force that pulls an adhesive apart by separating one flexible surface and one rigid surface.

4. Effect of temperature on joint strength

The temperature range and definition for a high-temperature adhesive have generally depended on the needs of the design engineer at the time. High temperature adhesive is designed for bonding insulating materials together and for their bonding to metal structures operating under elevated temperatures.

Methods of modeling and experimental results are presented and it is shown that the variation of the adhesive mechanical properties with temperature is generally the most critical factor. As the glass transition temperature of the adhesive is approached, a dramatic change in properties occurs. Techniques to improve the joint behavior are proposed, especially the mixed adhesive joint concept where a low temperature adhesive and a high temperature adhesive are used together in a joint.

Adhesive exhibits good adhesion to metal, glass and all types of refractory products and materials.

Experimental results of adhesive joints as a function of temperature generally show a decrease in strength at low and high temperatures. At low temperatures, the cause is the high thermal stresses whereas at high temperatures, it is the low adhesive strength. To increase the temperature range of application of adhesive joints, a joint with two adhesives along the overlap is proposed. A HTA in the middle of the overlap is the load bearing adhesive at high temperatures whereas a LTA at the ends of the overlap protects the HTA at low temperatures. This idea is supported with analytical, numerical and experimental results.

The change of adhesive properties with temperature is the main challenge to face for low and high temperature applications.

The temperature stresses may be important and they must be taken into account, especially those due to different CTEs. However, due to the polymeric nature of adhesives, the variation of the mechanical properties of the adhesives (stress-strain curve and toughness) with temperature may be the most important factor to consider.

For high temperature applications (above 200ºC), the adhesives used are often either bismaleimides or polyimides, and are generally supplied as films. Film adhesives can be supported, i.e. with a carrier, or be unsupported, i.e. without a carrier. In general, carriers can be of nylon, polyester or glass (usually preferable at high temperatures) fibres and their structure can be nonwoven, woven or knitted. Although polyimides have good high temperature properties, bismaleimides offer advantages such as the fact that they do not generate volatiles during cure, thus resulting in less porosity. When temperatures higher than 300ºC are desired, ceramic adhesives may be used . They are based in inorganic binding compounds such as alkali silicates and metal phosphates. Carbon, silica, magnesia, or zirconia powder is commonly used as fillers. The adhesives are usually available in two part systems, in which one of the components is the binder and the other is the filler. Joining these two components results in a slurry that must be quickly applied to the substrate. The assembly must then be cured, which requires temperatures between 260 and 1000ºC. The different combinations between binder and filler can be optimised to more closely match the CTE of the substrate and reduce the stresses during heating and cooling phases. Currently used ceramic adhesives do not offer a hermetic connection or high degree of reliability. Their reliability and strength is below that offered by high performance ceramics, and this can result in cracking under thermal stresses. The use of these adhesives is therefore a very complex proposition and requires careful design.

For aerospace applications, not only do extremely high temperatures have to be considered but sometimes extremely low temperatures as well. When in orbit, the outside temperature is of the order of -80ºC. Adhesives for cryogenic engines should maintain their mechanical performance to temperatures as low as -200ºC. The high temperature adhesives can be used at low temperatures. However, they are very sensitive to defects due to their brittle nature. It is more appropriate to use rubber-like adhesives such as polyurethanes or silicones. Most conventional sealants, such as polysulphides, flexible epoxies, silicones, polyurethanes and toughened acrylics are flexible enough for use at intermediate low temperatures to -30°C. Aerospace applications for these materials include their use as sealants for fuel tanks or for bonding the ceramic tiles of the space shuttle.

The mechanical properties of an adhesive depend on the operational temperature in relation to the glass transition point. As the temperature decreases below the glass transition temperature (T_g), the modulus and strength increase while the ductility decreases. At T_g there is a rapid reduction in the modulus and strength as the temperature increases, and the adhesive can no longer carry a substantial load. The knowledge of T_g is therefore very important. T_g can be determined by various methods such as dynamic mechanical thermal analysis (DMTA), thermo mechanical analysis (TMA) or differential scanning calorimetry (DSC). The results obtained by da Silva and Adams (2005) from a DMTA type apparatus where the modulus and the damping as a function of temperature are given for an epoxy adhesive (Hysol 9359.3, Loctite Aerospace). The high damping and modulus drop is clearly seen in the vicinity of T_g.

The effect of temperature from -55 to 200ºC on the shear stress – shear strain properties of an epoxy adhesive (Supreme 10HT, Master Bond) and a bismaleimide adhesive (Redux 326, Hexcel Composites). The bismaleimide adhesive has a maximum service temperature, according to the manufacturer, of 230°C for long periods and 270°C for short periods. The epoxy have a T_g close to 100°C. The bismaleimide adhesive strength decreases with temperature but its ductility increases, which can be an advantage when used in a joint. Jensen et al. (1995) tested the tensile properties of a polyimide adhesive in the film form (without carrier) at 25, 150 and 177ºC. The tensile strength and tensile modulus decreased with temperature (48% and 39% respectively) whereas the elongation increased by 31%.

Dixon et al. (1994) tested in lap shear and peel an epoxy for high temperatures and a bismaleimide from –55 to 130ºC. Both contained a carrier. They found that the bismaleimide adhesive has a more consistent strength over the temperature range tested than does the epoxy. Dixon et al. (1994) found that failures begin with small cracks at the specimen ends and that catastrophic failure is often seen to occur through the support cloth. Moreover, the bismaleimide adhesive failed in such a way that at 20 and –55ºC, the support cloth was cleanly debonded, whereas at 130ºC the adhesive remained attached to the cloth fibres after fracture, probably due to an increase in adhesive ductility. More recent results on the influence of temperature on adhesive properties are presented Deb et al. (2008) using double lap shear tests.

Besides the stress-strain variation, the toughness of the adhesive is also an important factor to consider as this material property is increasingly being used for adhesive joints design (Curley et al., 2000; Wang et al., 2003; de Moura, 2008). Information on the critical strain energy release rate at low or high temperatures is more difficult to find in the literature than the stress-strain curves of the adhesives and this is certainly an area that needs to be investigated. The area under the stress-strain curve is an estimate of the toughness. As the temperature increases, the strength decreases but the ductility increases giving an additional plastic deformation at the crack tip, hence an increase in toughness. Fracture tests in mode I at cryogenic, room and high temperatures have indicated that (Kim et al., 2005; Melcher and Johnson, 2007). However, a rapid decrease in toughness is expected as the T_g of the adhesive is approached.

The instantaneous response of a polymer material above its T_g is a small fraction of its total response to the load, with an important time dependent component. The models used to represent this behaviour are based on arrangements of Hookean springs or Newtonian dashpots. If the time during which the stress is applied is small compared to the time of relaxation, the response is determined more by the spring than the dashpots. For a polymer at a temperature below its T_g, the time for relaxation is almost infinitely long and therefore the response is almost elastic. As the temperature increases and approaches T_g, the modulus starts to decline and the viscous component becomes increasingly important. Generally, structural adhesives are used at temperatures below the vitreous state of the adhesive.

Richardson et al. (2003) proposed and evaluated for an epoxy adhesive a multiaxial, temperature, and time-dependent failure criterion. The failure criterion has been shown to be applicable for temperatures ranging from -29ºC to 46ºC. An important aspect of viscoelasticity occurs when the polymers are cyclically deformed. Even at temperatures below T_g there is always a viscous component that leads to a loss of energy during the cyclic deformation. One advantage of this phenomenon is that it can induce a vibration damping. However, energy converted to heat increases the temperature. In theory, the rise in temperature may affect the modulus and strength but, in practice, a large interfacial area provides excellent conditions for the release of heat. In the case of joints between metal materials, the increase in temperature under dynamic deformation is negligible. This can not be assumed in the case of other substrates, such as composites with carbon or glass fibres.

As seen above, thermal loads are a particular challenging condition for adhesives. The modifications in mechanical properties can be permanent or temporary. The ageing mechanism can be divided in physical and chemical. The physical ageing, which consists in a progressive loss of the free volume, can be recovered by heating the adhesive above the T_g and quenching (Parvatareddy et al., 1998). However, when the chemistry of the adhesive is altered after a stage at high temperatures, the damage is permanent. This aspect is particularly important and the service temperature of the structure should never go beyond the chemical degradation temperature of the adhesive.

There are cases, however, where the chemical changes are used as an advantage. Wang et al. (2006) have shown that the residue derived from the carbonization of organic synthetic resin possesses excellent thermal-physical properties.

A full study of the effects of temperature on joints must cover shrinkage of the adhesive, differential coefficients of thermal expansion and variation of adhesive mechanical properties with temperature such as the stress-strain curve and the toughness. [7]

5. Thermocouple description

Thermoelectric temperature measurement are made with thermocouples, devices that convert a temperature difference into an electromotive force called the Seebeck voltage,[8] and are widely used in science and industry. Thermocouples are based on the principle that when two dissimilar metals are joined a predictable voltage will be generated that relates to the difference in temperature between the measuring junction and the reference junction (connection to the measuring device).[9] They are "simple" rugged, need no batteries, measure over very wide temperature ranges and more. They have their quirks, too, like everything else. The solution Thermocouple uses and problems lies in the details of a given application.[10]

Thermocouples are usually made from two dissimilar metal wires connected so that one junction is held at a reference temperature sensing device.

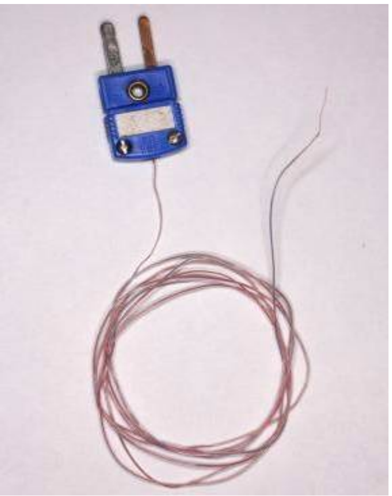

Figure 5. Thermocouple

Selecting the wire size used in the thermocouple sensor depends upon the application. Generally, when longer life is required for the higher temperatures, the larger size wires should be chosen. When sensitivity is the prime concern, the smaller sizes should be used. Since the effect of conduction of heat from the hot end of the thermocouple must be minimized, the thermocouple probe must have sufficient length. Unless there is sufficient immersion, readings will be low. It is suggested the thermocouple be immersed for a minimum distance equivalent to four times the outside diameter of a protection tube or well. Thermocouples should always be in a position to have a definite temperature relationship to the work load. Usually, the thermocouple should be located between the work load and the heat source and be located approximately 1/3 the distance from the work load to the heat source.[9]

Table 2. Thermocouple Reference Table (mV vs. Temperature)[9]

Thermocouple Type	Names of Materials	Useful Application Range
B	Platinum30% Rhodium (+) Platinum 6% Rhodium (-)	2500 -3100F 1370-1700C
C	W5Re Tungsten 5% Rhenium (+) W26Re Tungsten 26% Rhenium (-)	3000-4200F 1650-2315C
E	Chromel (+) Constantan (-)	200-1650F 95-900C

J	Iron (+) Constantan (-)	200-1400F 95-760C
K	Chromel (+) Alumel (-)	200-2300F 95-1260C
N	Nicrosil (+) Nisil (-)	1200-2300F 650-1260C
R	Platinum 13% Rhodium (+) Platinum (-)	1600-2640F 870-1450C
S	Platinum 10% Rhodium (+) Platinum (-)	1800-2640F 980-1450C
T	Copper (+) Constantan (-)	-330-660F -200-350C

5.1 Thermocouple Circuit

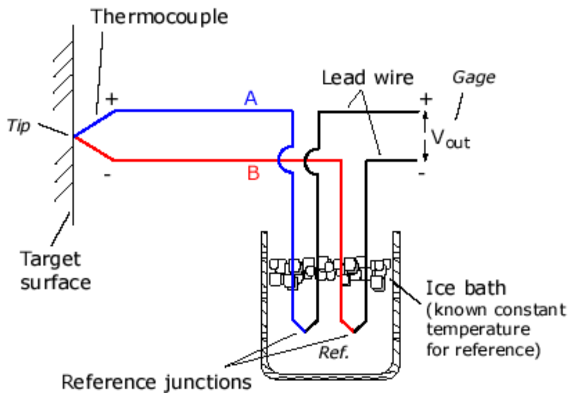

Figure 6. Thermocouple Circuit [11]

Suppose that the Seebeck coefficients of two dissimilar metallic materials, metal A and metal B, and the lead wires are S_A, S_B, and S_{Lead} respectively. All three Seebeck coefficients are functions of temperature. The voltage output V_{out} measured at the gage (see schematic above) is,

$$\begin{aligned}
V_{out} &= \int_{Gage}^{\mathrm{Re}\,f} S_{Lead}(T)\frac{dT}{dx}dx + \int_{\mathrm{Re}\,f}^{Tip} S_A(T)\frac{dT}{dx}dx + \int_{Tip}^{\mathrm{Re}\,f} S_B(T)\frac{dT}{dx}dx + \int_{\mathrm{Re}\,f}^{Gage} S_{Lead}(T)\frac{dT}{dx}dx \\
&= \int_{T_{\mathrm{Re}\,f}}^{T_{Tip}} S_A(T)dT + \int_{T_{Tip}}^{T_{\mathrm{Re}\,f}} S_B(T)dT \qquad (16) \\
&= \int_{T_{\mathrm{Re}\,f}}^{T_{Tip}} [S_A(T) - S_B(T)]dT
\end{aligned}$$

where T_{Ref} is the temperature at the reference point, T_{Tip} is the temperature at the probe tip. Note that mathematically the voltage induced by the temperature and/or material mismatch of the lead wires cancels, whereas in reality the lead wires will introduce noise into the circuit.

If the Seebeck coefficient functions of the two thermocouple wire materials are pre-calibrated and the reference temperature T_{Ref} is known (usually set by a 0°C ice bath), the temperature at the probe tip becomes the only unknown and can be directly related to the voltage readout.

If the Seebeck coefficients are nearly constant across the targeted temperature range, the integral in the above equation can be simplified, allowing one to solve directly for the temperature at the probe tip,

$$V_{out} = (S_A - S_B)(T_{Tip} - T_{\mathrm{Re}\,f}) \\ \rightarrow T_{Tip} = T_{\mathrm{Re}\,f} + \frac{V_{out}}{S_A - S_B} \quad , (17)$$

In practice, vendors will provide calibration functions for their products. These functions are usually high order polynomials and are calibrated with respect to a certain reference temperature, e.g., 0 °C (32 °F). Suppose that the coefficients of the calibration polynomials are a_0, a_1, a_2, ..., a_n. The temperature at the probe tip can then be related to the voltage output as,

$$T_{Tip} = a_o + a_1 V_{out} + a_2 V_{out}{}^2 + \ldots + a_n V_{out}{}^n \quad , (18)$$

Note that the above formula is effective only if the reference temperature T_{Ref} in the experiment is kept the same as the reference temperature specified on the data sheet. Furthermore, these coefficients are unit sensitive. Make sure to use the vendor-specified temperature unit when plugging in numbers.

Again, a thermocouple is a relative not absolute temperature sensor. In other words, a thermocouple requires a reference of known temperature which is provided by ice water in the above illustration.. Thus, common commercialized thermocouple often includes another temperature sensor, such as thermistor, to provide the reading of the reference (room/surrounding) temperature. [11]

6. Experimental programme

The objective was to develop an experimental device to simulate the ceramic tile shielding found in aerospace vehicles. The materials, geometries, manufacture and test are described here.

6.1 Adhesive description

The adhesive used was a high temperature epoxy adhesive, Araldite AV 138 M with Hardener HV 998. When fully cured the adhesive has excellent performance at elevated temperatures and has high chemical resistance. It is suitable for bonding a wide variety of metals and ceramics. It is widely used in many industrial applications where resistance to aggressive or warm environments is required. The low out gassing makes this product suitable for aerospace applications.

The key properties for this adhesive are,

- Low out gassing, volatile loss
- Excellent chemical resistance
- Temperature resistance to 120°C
- Cures at temperatures down to 5°C
- Thixotropic, gap filling paste [12]

Some properties given by the manufacturer are presented in Table 3.

Table 3. Typical product data of the adhesive [12]

Property	***AV 138 M***	***HV 998***	***Mixed adhesive***
Colour (visual)	beige	grey	grey
Specific gravity	ca. 1.7	ca. 1.7	ca. 1.7
Viscosity (Pas)	thixotropic	thixotropic	thixotropic
Pot Life (100 gm at 25°C)	-	-	35 mins

The mix ratio is 100g Araldite AV138 to 40 g of Hardener HV 998.The curing times at each temperature are presented in Table 4.

Table 4. Curing times of the adhesive [12]

Temperature	°C	10	15	23	40	60	80	100
Cure time	hours	48	36	24	16	1	-	-
Cure time	minutes	-	-	-	-	-	15	10
LSS at 23°C	N/mm^2	10	11	13	14	15	16	18

6.2. Substrates

6.2.1 Aluminum

The aluminum plate shown in Figure 20 has excellent ductile property. Aluminum is one of the lightest available commercial metals with a density approximately one third that of steel or copper. Many applications require the extreme versatility which only aluminum possesses. The plate used has 14 cm of length, 8 cm of width and a 1mm thickness.

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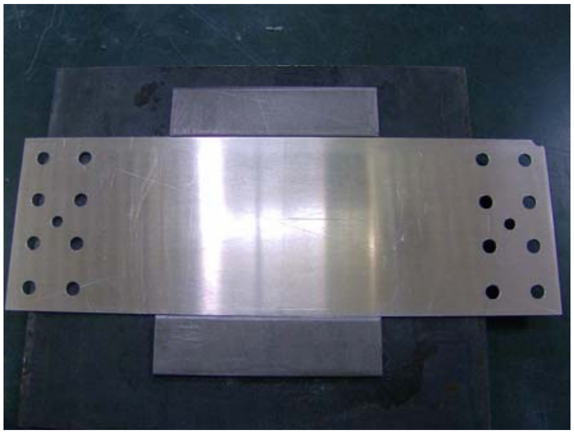


Figure 7. Image of the aluminum plate

6.2.2 Ceramics

Ceramic plates are widely used in reaction tanks, towers, troughs and ponds in metallurgical and chemical processes as equipment linings end bonding high temperature resistance. They are the special materials that can resistant high temperature, fast cooling and strong acid corrosion. The plate used has 14 cm of length, 9 cm width and a 5cm thickness. The original ceramic plate is bigger, and to obtain a plate of the intended size cutting is required. The original plate should be measured, and then lines are drawn to define the location of the cuts.

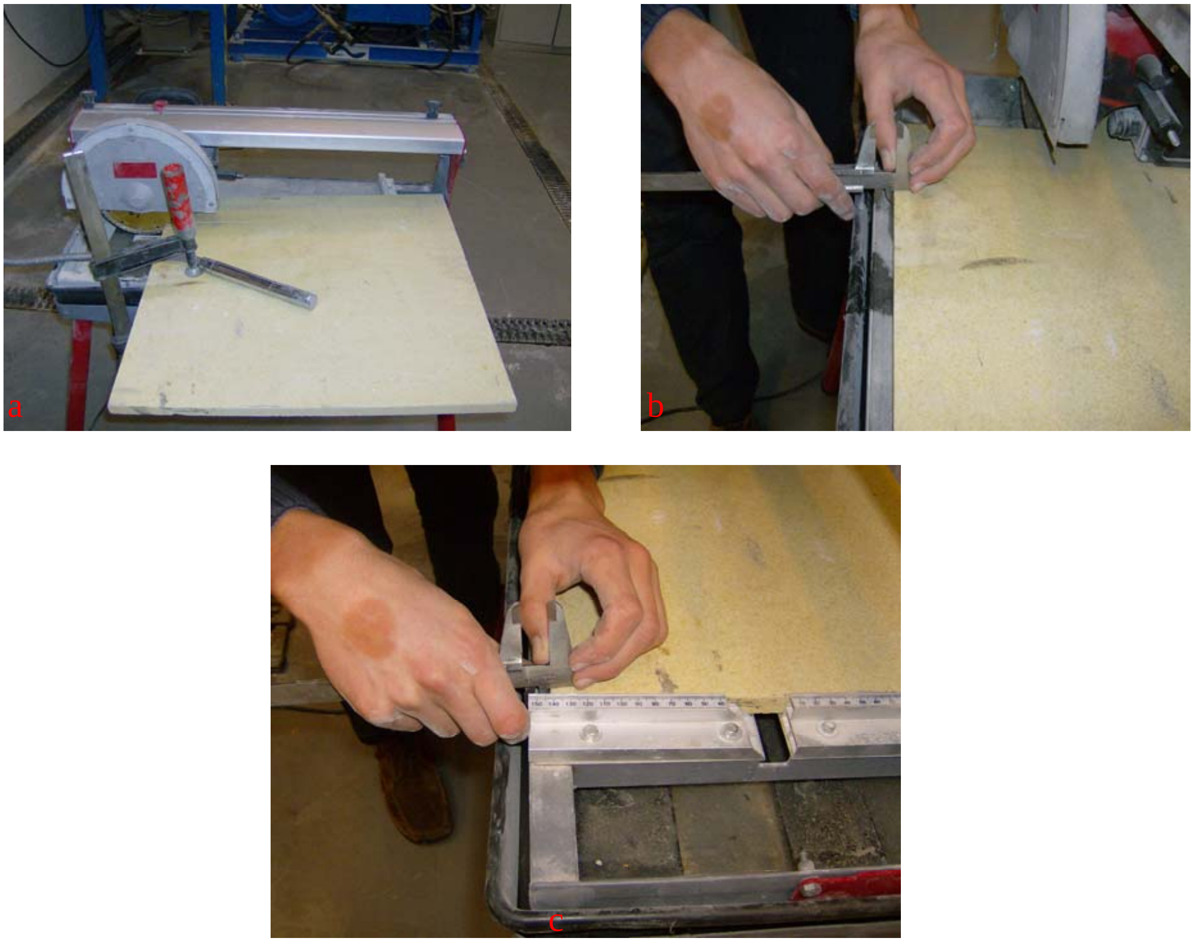


Figure 8. First step for cutting ceramics

During the ceramic cutting, water must be constantly applied to the blade to avoid overheating and blade damage.

Figure 9. The second stage of ceramic cutting.

In figure 8 see the ceramics cutting process stages. In a) one piece was cut to 14 cm length and 9 cm width; in b) a plate with width 14 cm was exactly as needed, to cut many pieces; in the last two c and d are representing how the measurement and final cut of the pieces that we needed was performed.

6.3 Thermocouple preparation

One of the most common tools that researchers use to measure temperatures of objects or organisms is the electronic thermocouple. The thermocouple readers are quite durable and last for years. On the other hand, the thermocouple wires eventually break after lots of usage. A typical T-type thermocouple was used with a “miniature” connector (figure 12).

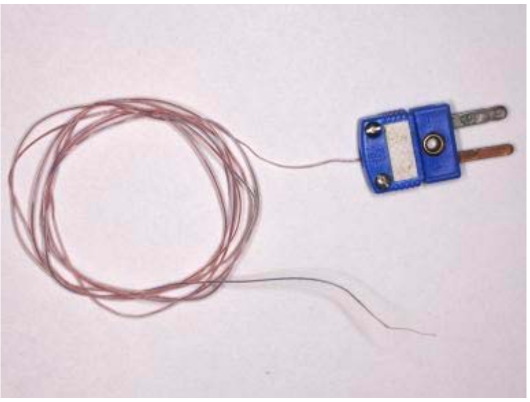


Figure 10. Thermocouple R-type

To reduce the effect of wire on the temperature distribution in the joint, a very thin wire was used. First thing to do is the cut of a thread of thermocouple, and separate the thread in two parts. The leads need to be pulled out of the insulation. A razor was used to pull perform this. Special care is needed to avoid cutting the metal wire itself, the cut should be enough just to take off the clear insulation and the colored insulation, leaving the bare wires intact. The excess insulation should also be cut so that things are neat and tidy. This process is done at both ends of thread (yarn).

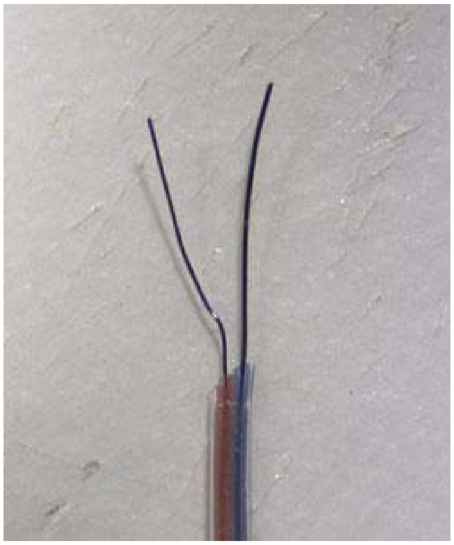


Figure 11. Picture of the yarn split in two parts

After splitting the yarn, a pair of tweezers is used to twist the two leads together.This provides a bit of mechanical strength to the joint, and makes the soldering easier. The soldering is performed in a bath of mercury, trough which a high voltage electrical current is run.

Figure 12. Picture of the soldering.

In the other side of the yarn, 1 cm of bare wire must be linked to connector. The cover on the connector end is unscrewed and two screw terminals are visible. On a T-type connector, one terminal will be copper colored and the other will be silver colored. They each have a screw in them, which is used to attach the thermocouple wires. The thermocouple wire is placed under one end and tightened. The rubber grips the thermocouple lead without cutting it, providing a measure of strain relief for the terminal connections inside the housing.

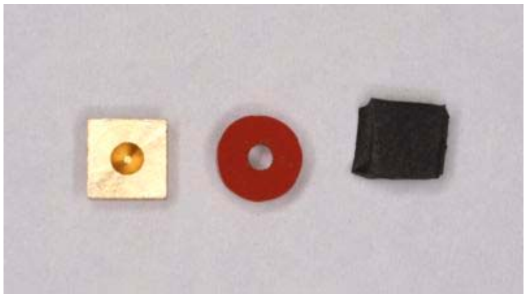
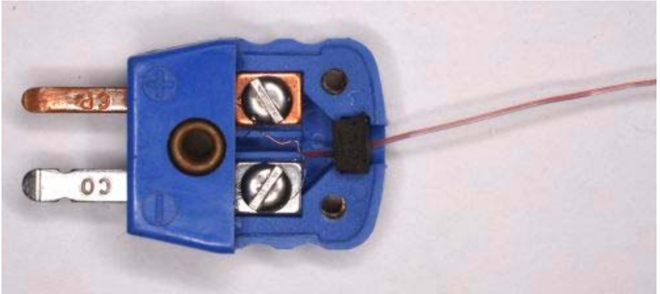


Figure 13. Rubber grips



The wire color is important: the copper colored wire needs to be screwed onto the copper-colored terminal, while the silver constantan wire needs to be screwed onto the silver terminal. With the yarn still taped down, the connector should be positioned so that the bare portion of the wire will sit completely inside the connector when closed. Finally, using the tweezers, thread around the screw is closed.

Figure 14. The yarn shown linked to connector.

After this assembly process is terminated, the completed thermocouple is tested with a multimeter/thermometer to ensure that it is working correctly.

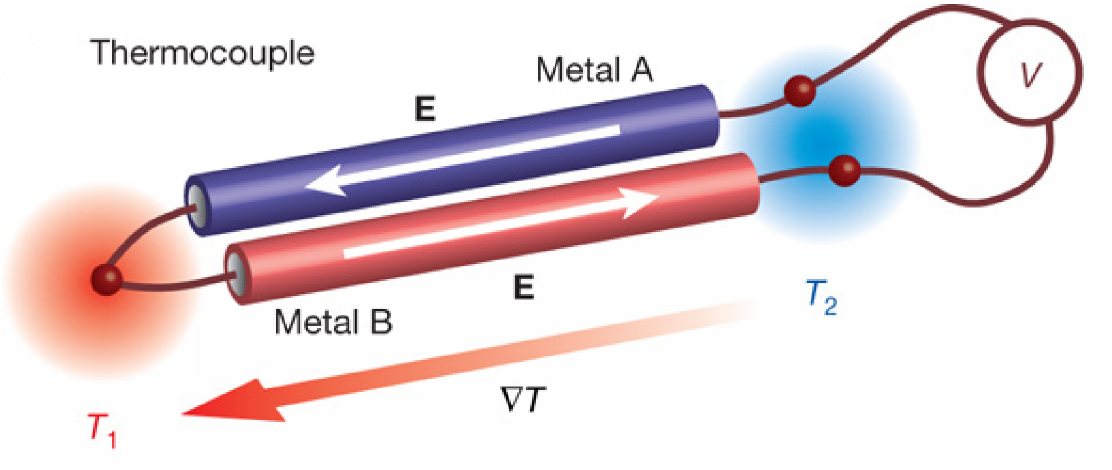
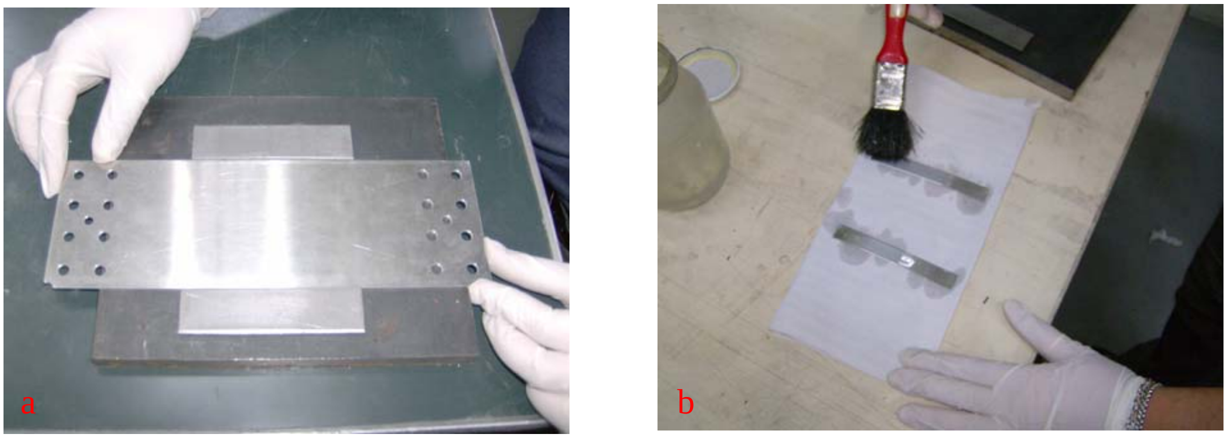


Figure 15. Testing of thermocouple

6.4 Joint assembly manufacture

Required for this procedure are: a ceramic plate, adhesive Araldite AV 138 M with Hardener HV 998, an aluminum plate and thermocouple to measure temperature. A base was obtained for the aluminum plate (Figure 21a) so that the pieces do not move while the adhesive is curing. In Figure 21b 2 spacers are shown, their thickness is 1 mm. These spacers are used to give the adhesive the desired thickness. These spacers are first cleaned with acetone and after that will mould release agent (Frekote 770-NC) (Figure 21c) is used, this procedure is done three times and should dry well the spacers. This procedure should also be be performed for the zone between the adhesive ends section and the base that supports the aluminium plate (Figure 21 d).



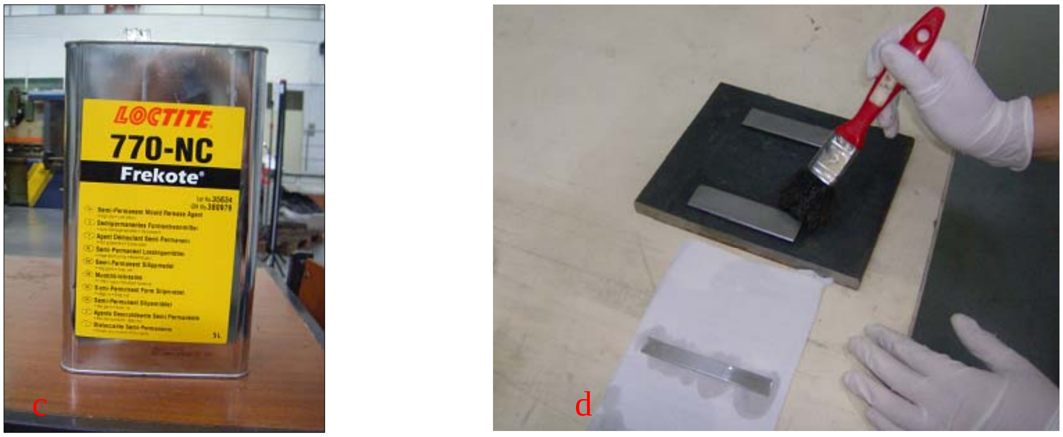


Figure 16. First step of the specimen preparation

Next step consists in cleaning the aluminum plate and ceramic (Figure 22a), After this is done, thermocouples are glued to the center of the surface of the ceramic (Figure 22b). In the aluminum plate the thermocouple must also be at the center. This is done by drawing two diagonal lines on the width of the plate (Figure 22b. At the intersection we glue the adhesive with cyanoacrilate (super-glue)

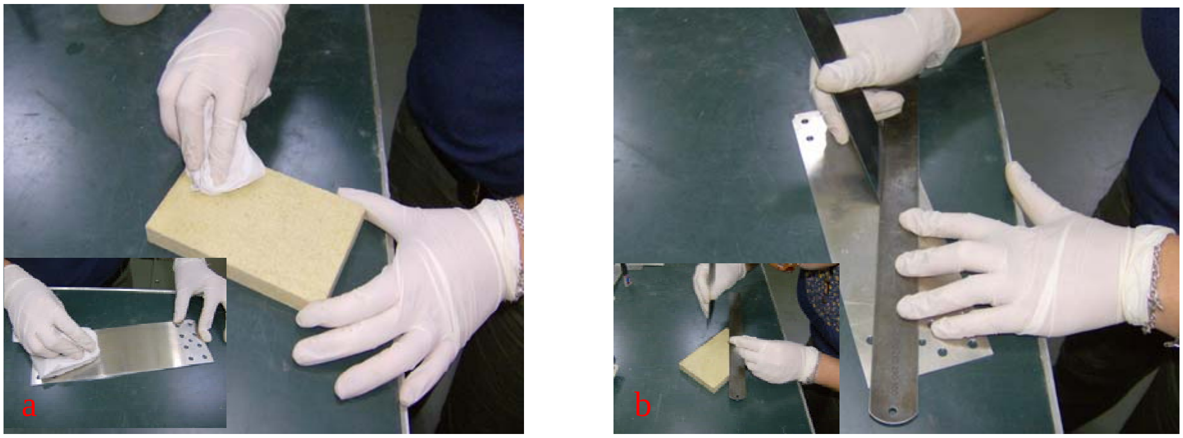


Figure 17. Cleaning the surface and marking the center.

Aluminum plate will be fixed in the base, and we apply spacers on the surface of aluminum plates, so that they don't touch the surface reserved for the adhesive. (Figure 23).

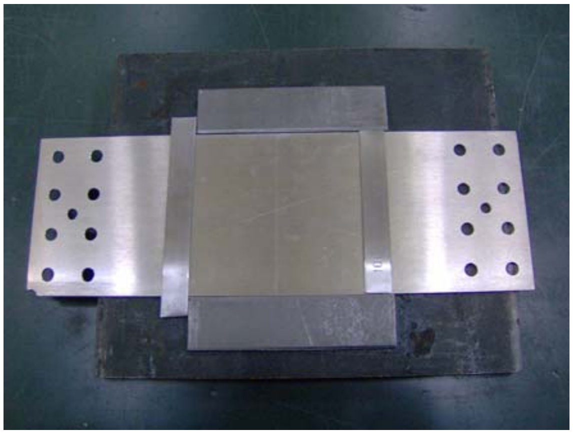


Figure 18. The area where adhesive will be appliIn the center of the aluminum plate there is the glued thread of the thermocouple (Figure 24). The same is valid for the ceramic plate. Each of these thermocouples will allow to register the internal adhesive temperature during the tests.

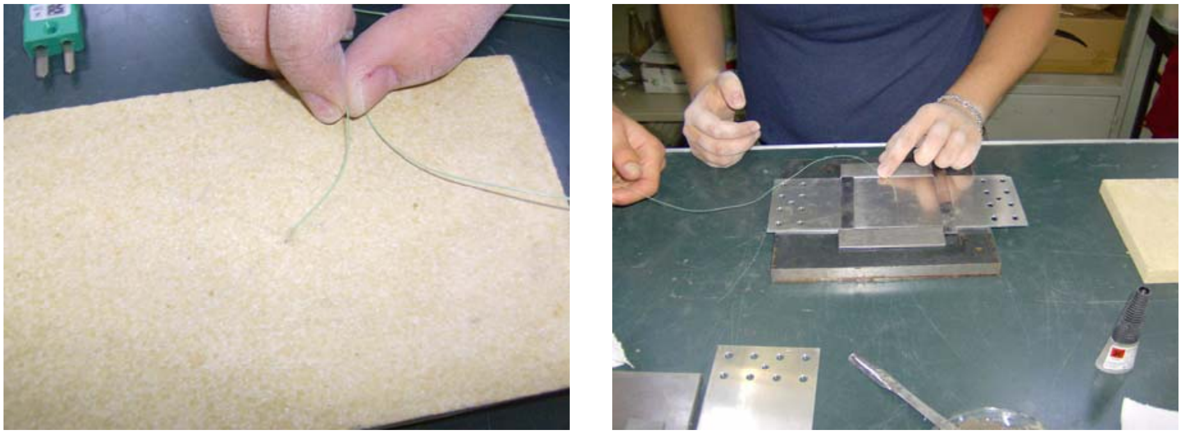


Figure 19. Putting the yarn of the thermocouple in the center plate

Next step is the adhesive mixing. Usually the adhesives sit in the fridge at 5-10 degrees, but before the test is done, the adhesive must be warmed to ambient temperature. This mixture has two components Araldite AV 138 M with Hardener HV 998 (Figure 25a). This type of adhesive provides a good resistance to high temperatures (120 ° C). The mixture of components resin / hardener it is a ratio of 100/40 respectively. 20g by Araldite AV 138 M and 8g of Hardener HV 998. To make this mix a clock glass was placed in the digital scale with spatula (Figure 25b). The adhesive was applied on the surface with a spatula but care must be had to ensure the perfect homogeneity of the resin with hardener.

Table 5. The mixture of components Araldite AV 138 M with Hardener HV 998

Mix ration	**Parts by weight**	**Parts by volume**
Araldite AV 138 M	100	100
Hardener HV 998	40	40

Figure 20. Components Araldite AV 138 M with Hardener HV 998

After the adhesive was homogenized, it was applied in the square drawn on the aluminium plate. It was applied from the outside to the inwards, so that no blank is left. At the same time care must be had with thread of the thermocouple so that the thread is not severed.

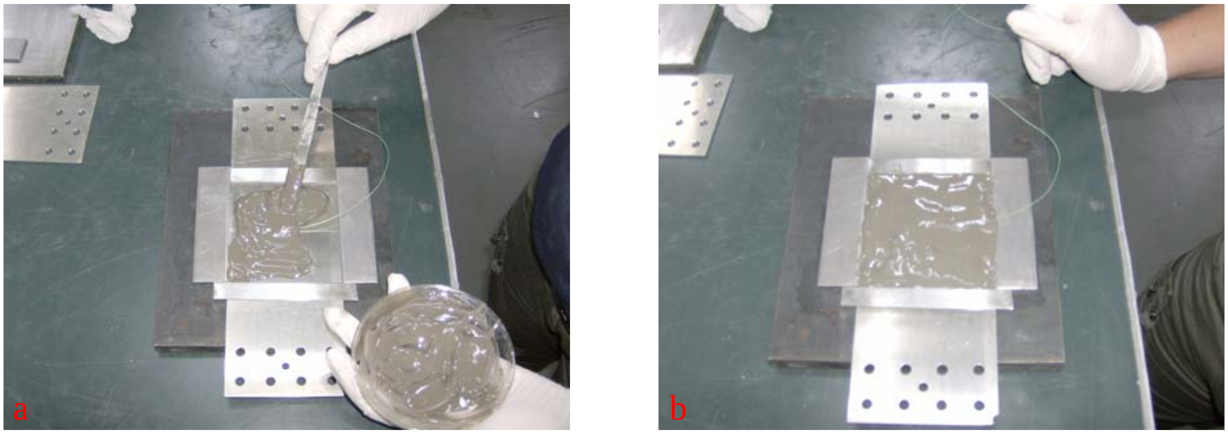


Figure 21. Applying the adhesive

The last step is the application of the adhesive in the ceramic plates. The application must be carefully done so that the ceramic plate is set exactly in the centre of the aluminum plate. Again, care must also be had to avoid damaging the thermocouple wire.

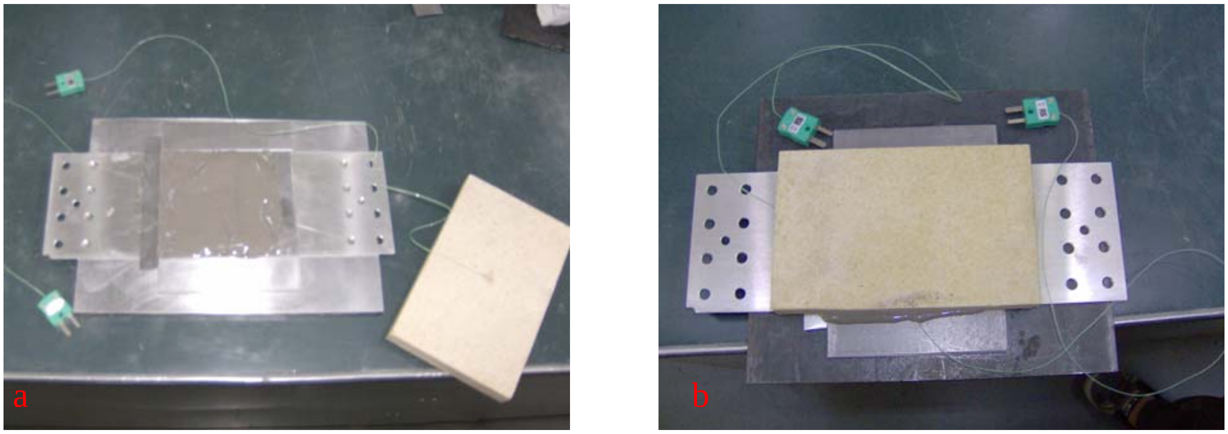


Figure 22. Picture the last step

This specimen must sit in the oven for 3 days at a temperature of 25ºC.After the cure is finished, a new thermocouple is bonded to the center of the rear of the aluminum specimen. The thermocouples are then all tested, using a hot air blower and multimeter/thermometer. If all thermocouples respond positively to the heat, the specimen is considered good.

6.5 Test set-up

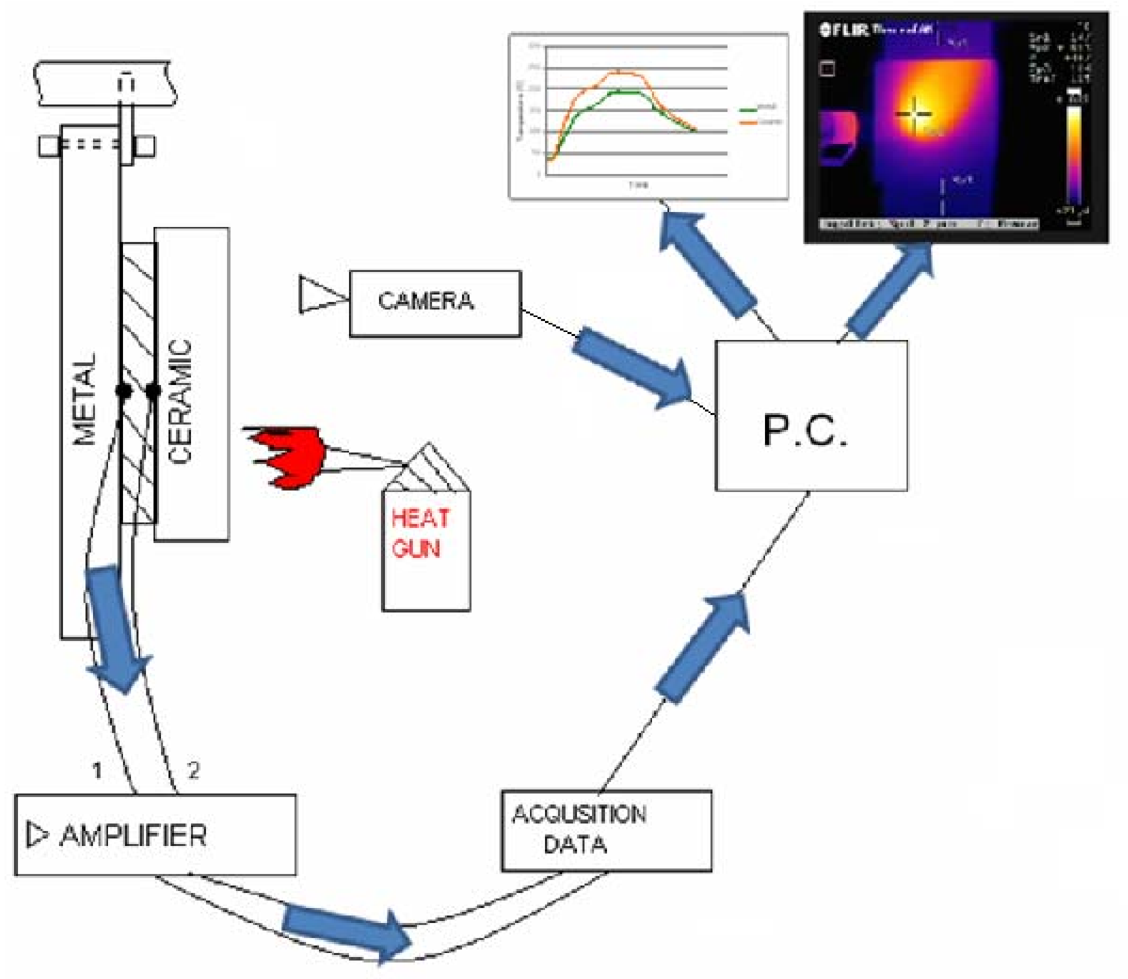


Figure 23. Test set-up

This test setup uses a structural frame to hold the ceramic specimen which will be subjected to a direct flame from a gas burner until it reaches 600°C of temperature at surface.

An infrared camera is used to capture the stream of images [movie] with the temperature field. This field is analyzed with a specific software, allowing to observe the temperature. This movie is recorded in a laptop.

The thermocouples attached to the inner surface are then connected to an amplifier and to a data acquisition board registering the temperatures in a laptop. These temperatures are plotted in a graph for better understanding of the phenomena happening in between the two surfaces and the adhesive.

7. Results and discussion

For the final test an heat source and propane torch were , to obtain a full power on the ceramics surface. In this test an infra-red camera measured the maximum surface temperature.

In the previous figure a schematic image of the specimen is shown. T_1 represents the temperature measure from the first thermocouple, T_2 represents the temperature measure from the second thermocouple, and T_3 represents the temperature measured from the camera with infra-red.

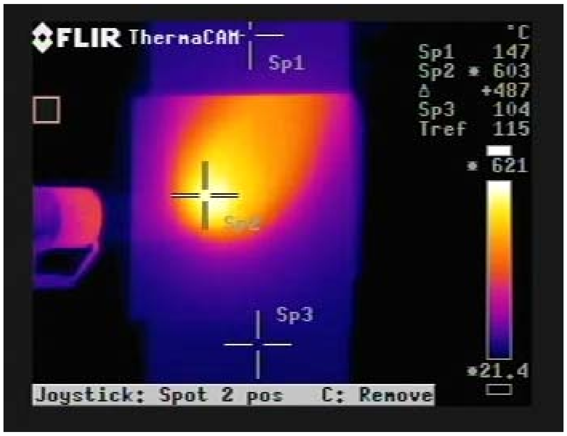


Figure 24. Representation of the temperature on the surface of the ceramic (T_3)

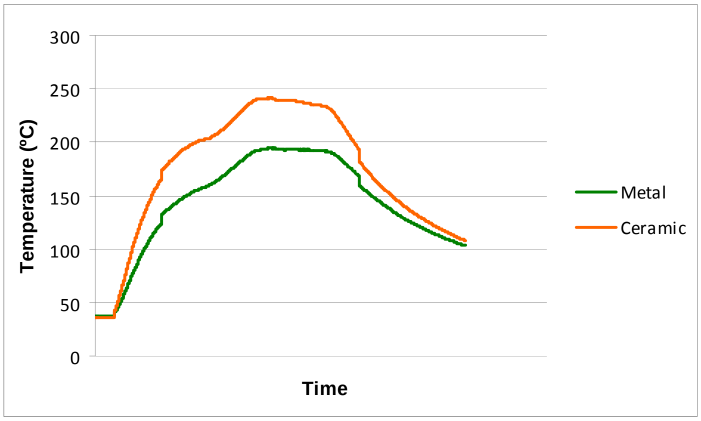


Figure 25. Representation of temperature measured by the thermocouple T_1 and T_2

Table 6. Representation of the temperature measured on the specimen

	Maxim	Time to maximum temperature	Difference between maximum and minimum temperature
T_1	194ºC	801.96 s	870.84 s
T_2	241ºC	801.14 s	878.22 s
T_3	603ºC	570 s	-

8. Conclusion

- Infrared camera proves that temperature distribution is reasonably uniform.
- Ceramic used is good enough to guarantee that temperature in the bondline is what is found in practice.
- Test set up simulates relatively well the reality.
- Future work will combine mechanical and thermal loading of the specimen simultaneously.

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