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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE

No. 989

SURVEY OF ADHESIVES AND ADHESION

By R. C. Rinker and G. M. Kline
National Bureau of Standards

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SUMMARY

The development of adhesives for bonding metal, wood, rubber, and plastic parts on aircraft has been largely empirical. A better understanding of the physical and chemical forces involved in adhesion is needed for further rational improvement of bonding materials for use in aircraft construction.

A research project has been undertaken at the National Bureau of Standards under the sponsorship of the National Advisory Committee for Aeronautics to obtain information on the strengths of bonds between different chemical types of adhesives and adherends. The present report, which covers a survey of the present knowledge on the nature of adhesion, has been prepared as the initial step in this investigation.

The scope of the survey is indicated by the following outline:

I. Theoretical aspects of adhesion.
  A. Intermolecular and interatomic forces. The four types of bonds involved in the attractive forces holding matter together are reviewed.
    1. Electrostatic or polar bonds.
    2. Covalent bonds.
      a. True covalent bonds.
      b. Coordinate covalent bonds.
    3. Metallic bonds.
    4. Van der Waals forces.

  B. The nature of cohesion. The effects of the intermolecular and interatomic forces as measured by the physical behavior of substances held together by each type are discussed.

1. Crystalline matter.
   a. Covalent crystals.
   b. Ionic crystals.
   c. Coordinate covalent crystals.
   d. Crystals containing hydrogen bonds.
   e. Metallic crystals.
   f. Van der Waals forces in crystals.
2. Noncrystalline matter.
   a. Metals.
   b. Glass.
   c. Wood.
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I. THEORETICAL ASPECTS OF ADHESION

A. INTERMOLECULAR AND INTERATOMIC FORCES (REFERENCES 1 AND 2)

All matter is composed of discrete particles or atoms which are held together by electrical forces of attraction. These forces operate between the positively charged atomic nuclei and the negatively charged orbital electrons. By studying the physical and chemical properties of compounds of atoms it has been possible to recognize certain types of these attractions as differing from others. These differences, however, are not distinct and only in extreme cases is a bond a manifestation of a pure type. It is much closer to the truth to think of these bonds as having well defined properties, but to bear in mind that the transition from one type to another is not sharp but may be very gradual. When a bond is formed it does not mean that all the forces between the electrons and nuclei of the two participating atoms have been neutralized, but rather that a force concentration has been set up in a certain direction. Residual energy is always present.

In explaining chemical and physical phenomena it is convenient to recognize four general types of bonds:

Electrostatic

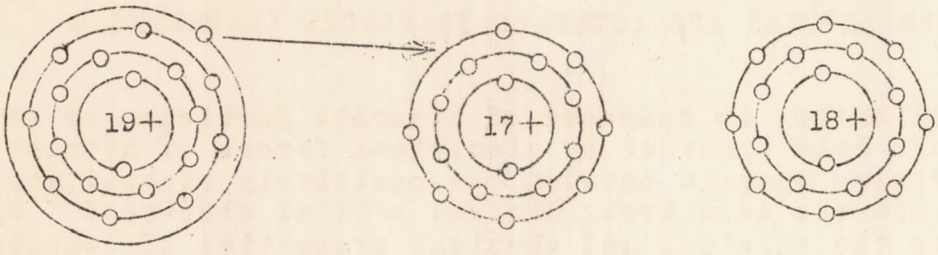
Covalent

Metallic

Residual bonding attraction forces, commonly known as Van der Waals forces

1. Electrostatic or Polar Bonds

Kossel (reference 3) in 1916 showed that an element immediately preceding a rare gas in the Periodic Table is strongly electronegative; whereas one immediately following it is strongly electropositive. The rare gases, on the other hand, are remarkably inert. He concluded that this behavior was due to a tendency on the part of the reactive elements to change their configurations in order to conform with those of the rare gases. Thus, an atom of potassium combines with one of chlorine by means of an electron transfer which results in the configuration of argon for each.



Potassium Chlorine Argon

By means of this mutual claim on the shared electron the two ions will remain closely associated with one another as potassium chloride. This association can be interfered with, however, as by solution in water, in which case the potassium chloride will dissociate into chlorine as a negative ion and potassium as a positive ion. If the water is removed by evaporation, these ions will aline themselves to form a crystal lattice. In such a solid the attractive forces between the oppositely charged ions except those on the surface will be satisfied. However, in an amorphous solid some of the attractive forces will not be satisfied because of randomness of distribution of the ions.

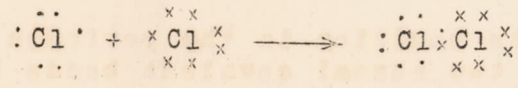
2. Covalent Bonds

Compounds which are formed of atoms removed by three places from the rare gases in the periodic table show a greatly lowered tendency to ionize. Thus, ammonia and phosphine are only very slightly ionized. In many molecules it is difficult or impossible to determine which atom is positive and which negative - for example, sulfur dioxide and carbon dioxide and, still more strikingly, the diatomic gases such as hydrogen and chlorine. These substances do not ionize in solution.

In 1916, G. N. Lewis (reference 4) proposed that in substances of this type it is possible for two atoms to share electrons in such a way as to account for the stability of each in attaining the rare gas configuration. Langmuir suggested that this type of linkage be termed "covalent."

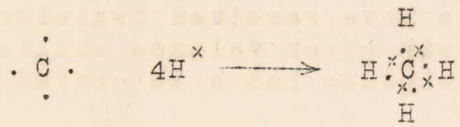
a. True covalent bonds.- In a diatomic molecule such as chlorine gas, each atom requires an electron to complete its

outer shell. In each atom the positive attraction of the nucleus is equally great on each of the two negative charges of the shared pair. This may be represented as follows:

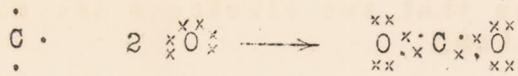


Such bonding is the most common form in organic molecules and accounts for their complexities and magnitudes. Thus, carbon has an outer shell of four electrons which it will share with as many atoms as will contribute toward a stable structure of eight.

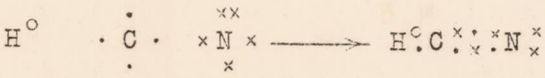
1. Monavalent atoms: Carbon may share an electron with each of four hydrogen atoms to give the stable gas methane.



2. Divalent atoms: Carbon may share two electrons with each of two oxygen atoms to give carbon dioxide.



3. Trivalent atoms: Carbon may share three electrons with one nitrogen atom in a covalent bond, such as in hydrogen cyanide.



Carbon also has the ability to form covalent bonds with itself an almost infinite number of times. It is well established that the structure of crystalline carbon (diamond) is formed by single covalent bonds which accounts for its very great stability.

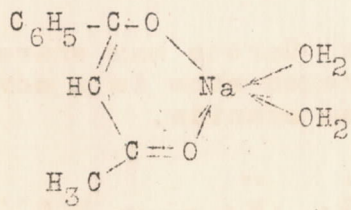
b. Coordinate covalent bonds.- Compounds exist the properties of which do not fit into any of the previously discussed categories. In 1892 Werner (reference 5) advanced a theory to account for them. These substances, formerly

shelved under the name "molecular compounds," appear to result from the union of two or more saturated molecules. They are also apparently independent of the groups in the periodic table to which the atoms involved belong.

Because of its position in the periodic table sulfur can form only the two normal covalent bonds because this brings about the completion of its full complement of eight electrons. Of these eight, two are shared with other atoms, as in dimethyl sulfide. This compound can, however, be oxidized as follows:

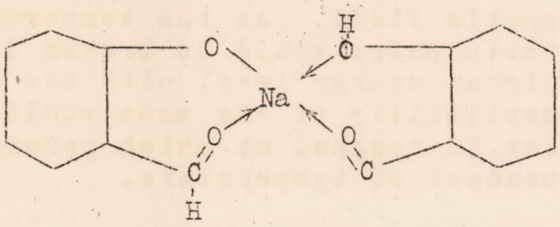
The two oxygen atoms have received two electrons each from the sulfur; thus their outer valence shells are complete, whereas the sulfur already had a saturated shell.

This same mechanism is used to explain another phenomenon occurring in organic molecules: namely, the chelate compounds. The peculiar properties of the hydrate of the sodium derivative of benzoyl acetone indicate that this substance may be represented by the following formula in which the arrow indicates that two electrons are contributed by each such oxygen atom.

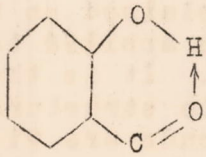


If this water were present as water of crystallization, the compound should have the same chemical properties as the anhydrous salt. The hydrate, however, dissolves in toluene; whereas the anhydrous salt is insoluble.

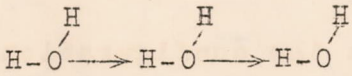
A similar example is given by the behavior of ortho-substituted phenols such as salicylaldehyde which form chelation compounds readily.



Further evidence indicates that the phenolic hydrogen in salicylaldehyde is itself linked to the oxygen of the aldehyde group in this same manner:



This leads to a very important case of this type of bonding: namely, the formation of hydrogen bridges between molecules. Many substances containing hydroxyl groups exhibit this phenomenon and even water has been shown to have a trimeric structure in ice.



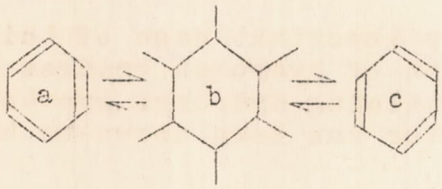
The fact that most molecularly associated substances contain hydroxyl or related groups leads to the conclusion that this type of bond may play a very important role in cohesive or adhesive phenomena.

3. Metallic Bonds

The metals which comprise the greatest number of elements in the periodic table owe their unique properties to a bond type which was not recognized until recently. Lorentz (reference 6) first advanced the theory that a pure metal consists of a crystalline arrangement of metallic cations with free electrons moving in the interstices. In 1927, Pauli (reference 7) postulated that these electrons exist in a continuous set of energy levels. At absolute zero they

occupy the most stable levels in pairs with oppositely opposed spins so that they would be unavailable for orientation in an applied magnetic field. As the temperature rises, however, these stable pairs would be broken down as one electron rises to a higher energy level with the result that the paramagnetic susceptibility of the mass would be raised until finally a point is reached at which paramagnetic susceptibility is independent of temperature.

If a simple metal such as the lithium crystal is considered, an arrangement of eight atoms is found at the corners of a cube. The distances of these atoms from one another satisfy the crystallographers' requirement for a single electronic bond. Other characteristics indicate the existence of double electron bonds as well. The stability of the structure may be explained on the basis of the theory of resonance which has been applied to many types of structures in organic chemistry. It is the same concept which explains the familiar Kekule structure of the benzene molecule in which the double bonds are visualized



as alternating between the configurations (a) and (c) with (b) as an intermediate.

Thus, any two atoms in a lithium crystal may be linked by two electrons or by one electron at a given instant. The single electron bonds are also used to account for the halides of boron having structures which could be explained in no other way. In effect the resonance between single and double electron bonds is a very specialized case of covalent bonds.

4. Van der Waals Forces

Up to this point the more potent forces of attraction between atomic nuclei and orbital electrons have been discussed. All these interactions may be thought of in terms of units of energy. These units, however, participate in maximum and not total exchanges. When a bond is formed, the positive and negative charges of the participating molecules

are not completely neutralized. There remain in many molecules residual energies which are very nearly of stable bond-forming magnitudes. These forces affect the melting and boiling points of many substances.

Matter composed of both electrons and protons always exerts attraction. A liquid will wet a solid surface to a degree dependent upon the magnitudes of the attractive or adhesive forces between the two substances and the cohesive forces within each. In the case of a drop of mercury on a glass surface, the cohesive forces within these two materials is much greater than the adhesion between them; consequently the mercury droplet almost forms a sphere, but there is sufficient attraction to distort this shape.

Many substances which will react at higher temperatures are attracted by Van der Waals forces at lower ones. It will be seen later that these attractive forces initiate reactions by virtue of the fact that their magnitudes are potent over greater distances than those of an electrostatic nature which are responsible for bond formation.

It was in recognition of these facts that Van der Waals presented his corrected version of the equation of state for gases and means for calculating the magnitudes of these residual attractive forces for all substances. It has been found that they are greater for the molecules of compounds than for molecules of elements or inert atoms, thus showing that unequal distribution accounts for a greater residual force field and consequently the more asymmetric the molecule the greater these values will be. This is a matter of great importance in explaining adhesive and cohesive properties.

B. THE NATURE OF COHESION

The mechanism whereby molecules or atoms in homogeneous matter are held together is known as cohesion. The physical state as well as the mechanical properties of the material depend upon the type and magnitude of its cohesive bonds. The operational effects of such bonds have been investigated by Lennard-Jones and coworkers (reference 8) by considering the case of an external particle approaching a crystalline surface. In such instances one of several phenomena may occur.

Chemical attraction.- If the particle is an ion, a direct electrostatic force will be set up between the charges

on the approaching ion and the ions on the surface of the crystal.

Polarization.

(a) Whether the particle is charged or not, an induced dipole will be set up in the atom or ion by the electric field of the crystal.

(b) If the particle is charged, it will polarize the ions in the crystal.

Attraction through Van der Waals forces.- Inasmuch as attraction exists even between two so-called neutral atoms (atoms of a rare gas), it is assumed that a similar attraction will be exerted between the crystal ions and the charged or uncharged particle, quite apart from the effect of their valency charges.

On the basis of this analysis, these investigators calculated these effects between neon and a charged particle, respectively, near the 100 plane (planes parallel to the face) of a sodium fluoride crystal; and between argon and a charged particle, respectively, near a potassium chloride crystal. These calculations showed that the electrostatic attractions fall off very rapidly as the distance from the surface increases, but are very powerful within a short optimum range. The Van der Waals attractions, on the other hand, have a much wider range, but are never as powerful as the electrostatic attraction. The polarization forces never become very great. These relationships are illustrated in figure 1.

These forces, with different degrees of magnitude, are responsible for the cohesion between ions, atoms, and molecules of all forms of matter. On the basis of this evidence, as well as the fundamental precepts of chemistry and physics, it is possible to ascribe points of strength and weakness to these forces in their cohesive capacity. The net effects of these forces determine the gross mechanical properties of all materials.

It must be realized, however, that the intrinsic strength which is indicated for materials is rarely achieved. When a test specimen of crystalline rock salt is broken in tension, the breaking load is 0.4 kilogram per square millimeter; whereas the calculated tensile strength is 200 kilograms per square millimeter. Joffe (reference 9) found,

however, that by surrounding the test specimen with hot water, loads as high as 160 kilograms per square millimeter could be obtained. He explained this on the ground that "premature" failure is due to the presence of submicroscopic scratches or fissures on the surface of the specimen, around which extremely high stresses may be concentrated. By removing these fissures as they were formed - that is, by continually dissolving away the surface - he was able to break the bonds along a plane of the crystal and thus approach the theoretical strength.

The presence of these minute cracks has been proved (reference 10) by sputtering metallic films on crystalline surfaces which results in fine lines of the metal. The reason for these cracks probably lies in the fact that ideal packing in crystals does not exist due to greater electrostatic forces within the substance than near the surface, thus strains are set up which cause the surface atoms to rearrange with relative ease under an applied load.

A brief consideration of several types of materials will serve to illustrate the behavior of the different cohesive forces.

1. Crystalline Matter

The criterion of crystallinity is the regularity of the arrangement of the atoms in a solid structure. Any atom in a crystal is separated from its neighbors by the same distances and at the same angles as any other atom of the same kind.

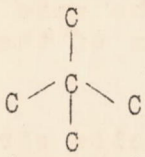
When a true crystal is subjected to a tensile stress it will rupture along the line of least resistance. The structure of crystals has been well established by X-ray and chemical studies. Hence, it is possible to identify the forces which were overcome along the cleavage plane.

a. Covalent crystals.- In the crystals of nonmetallic elements, single atoms share electrons with each other in pairs. Since neither atom exerts a greater influence on the bond than the other, a state of relative inertia exists. Two factors determine the effect of these bonds in providing cohesive stability, the bond distances, and the bond angles. The first of these factors determines the range over which the attractive force acts and how much it is interfered with by electrons closer to the nucleus of each atom. The second determines the configuration of the crystal.

It has been shown that carbon is capable of forming single, double, and even triple covalent bonds. That bond angle influences the structure is shown by the properties of diamond and graphite; whereas the effect of bond distance is illustrated by comparing diamond and crystalline silicon.

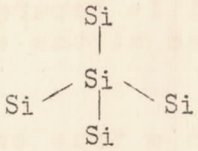
Diamond is the hardest and most inert of all crystals. It consists of carbon atoms joined to one another by primary covalent bonds; thus at the end of each carbon tetrahedron there is another carbon atom. There is relatively little strain in such a crystal and the distances between each atom and the next are relatively small. In the case of graphite, on the other hand, each carbon atom is attached to three of its neighbors instead of four, two through single bonds and one through a double bond. The angle of the double bond is quite different from that of the single. The result of this arrangement is the formation of layers of two dimensional plates which are rather loosely held by Van der Waals forces in the crystal. That these Van der Waals forces are strong enough to give some stability to the structure is to be expected from the fact that the energy of the double bond is less than twice that of a single bond.

In crystalline silicon in which the atoms have the same arrangement as in the diamond crystal, the bond distances are greater and the bond energies are consequently less; thus a weaker structure results.



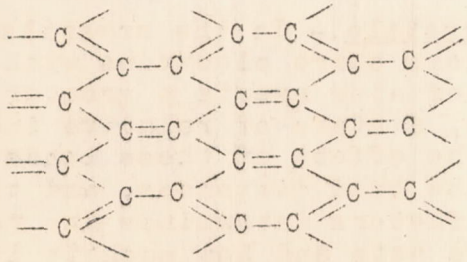
(Diamond)

Bond distance = 1.54 Å
Bond energy = 58.6 k.cal./mol.



(Silicon)

Bond distance = 2.34 Å
Bond energy = 42.5 k.cal./mol.



(Graphite)
(single bond) (double bond)

Bond distance = 1.54 Å and 1.34 Å
Bond energy = 58.6 and 100 k.cal./mol.

b. Ionic crystals.- Owing to the nature of ionic bond formation by the capture of an electron from an electropositive atom by an electronegative atom, the cohesive strengths of such crystals are weaker than those formed by covalent bonding. An ionic molecule splits into two fractions much more easily under the influence of chemical as well as physical forces. Thus, the solubilities are high and the strength properties low as compared to covalent substances. The same rules of bond distance and angles govern these strengths.

c. Coordinate covalent crystals.- The coordinate covalent bond has some of the properties of both ionic and covalent bonds. It has been called, in fact, the semipolar bond. The large groups of pure compound minerals are composed of complex crystals in which one molecular type is connected to another by the interlocking of their individual crystal lattices. Pauling (reference 1) has set forth several rules which apply to the types which are more ionic than covalent in character. These rules are based upon the concept that anions are coordinated at the corners of tetrahedral or polyhedral force distributions about each cation and that these polyhedra are joined at a corner, an edge, or a face. On this basis the formation and properties of such structures may be predicted as follows:

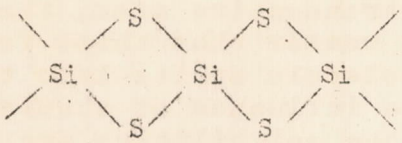
1. A coordinated polyhedron of anions is formed about each cation, the cation-anion distance being determined by the radius sum and the coordination number of the cation by the radius ratio.

2. In a stable ionic structure, the valence of each anion is exactly or nearly equal to the sum of the strengths of the electrostatic bonds to it from the adjacent cations.

3. The presence of shared edges, and especially of shared faces, in a coordinated structure, decreases its stability. This effect is pronounced for cations with large valence and small coordination number.

4. In a crystal containing different cations, those with large valence and small coordination number tend not to share polyhedron elements with one another.

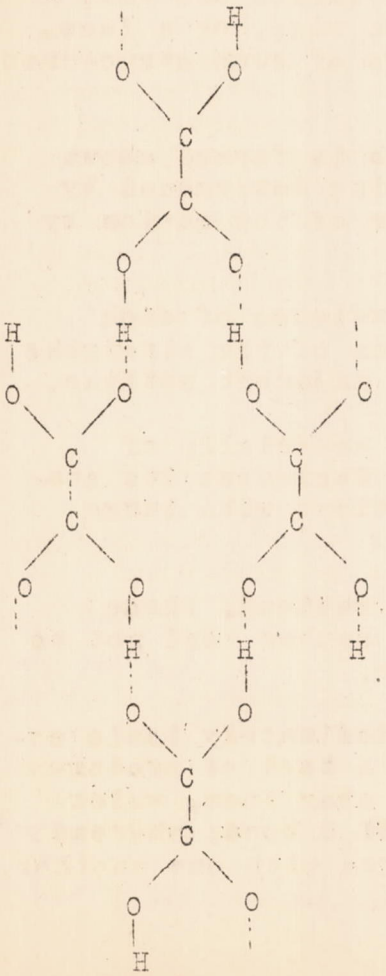
These rules are so applicable to predominately ionic coordinate bonds that variance from them is a test of predominant covalency. All the forms of silica obey them, which proves the partially ionic nature of the Si O bond; whereas with sulfur, the SiS_4 tetrahedra share edges with one another to give infinite chains.



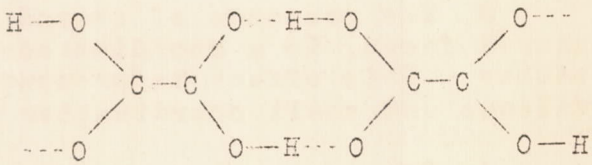
thus proving the Si-S bond to be essentially covalent.

d. Crystals containing hydrogen bonds.- The phenomenon of the hydrogen bond is responsible for the unusual properties of water. By analogy with other related compounds such as H_2Te, H_2Se, and H_2S, on the basis of decreasing molecular weights and Van der Waals forces, the melting and boiling points of water would be expected to be about -100° and -80° C, respectively.

A good example of C-O---H bonding in crystals is given by the two crystalline forms of oxalic acid. The alpha form of this substance consists of plates and the beta form of long chains.



alpha



beta

The plates and chains are interconnected through very weak Van der Waals forces.

e. Metal crystals.- The relationship between physical properties and bond distances and angles is the same in metal as in other crystals. If the atomic radii of the metallic elements are plotted against atomic number, it will be seen that a minimum occurs in each case between the sixth and tenth elements of each sequence. In this range, the greatest bond strength occurs and consequently the strongest metallic substances.

As the number of valence electrons per atom increases from 1 to 6, there is a corresponding increase in electron pairs effective in bonding the atoms together in the metal. Between the sixth and the tenth elements the number of electrons remains constant at about 6, but becomes smaller beyond the tenth. These relationships offer an explanation for the fact that properties dependent on cohesive forces, such as yield point and ultimate tensile strength and hardness reach maximimums between the sixth and tenth element in each sequence in the Periodic Table.

f. Van der Waals forces.- In crystalline substances the equilibrium of energies in the crystal lattice to give layers and threads (e.g., in graphite and asbestos, respectively) precludes the existence of very great bonding forces between these more or less saturated systems. That these forces do vary, however, is evidenced by the much greater interplanar cohesion for graphite than for oxalic acid or mica. These forces, therefore, depend on the crystalline configuration as well as the nature of the individual atoms involved.

2. Noncrystalline Matter

Most materials used for construction are not pure crystalline substances, but do have varying degrees of crystallinity. The cohesive behavior of amorphous substances may be explained on the basis of the more definite knowledge of crystal structure, inasmuch as the same forces hold them together.

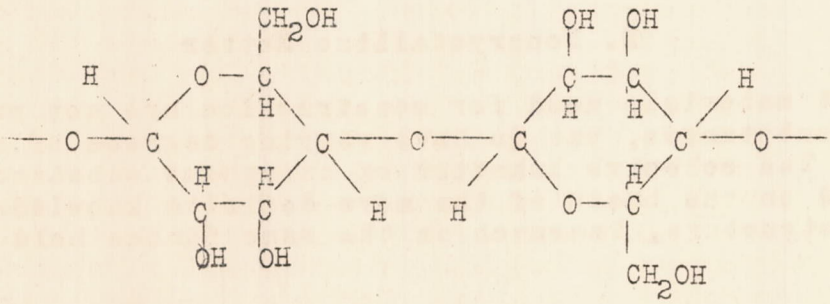
a. Metals.- Most metals consist of isolated regions of crystallinity surrounded by an amorphous medium. Alloys exhibit the properties of the component parts except where true intermetallic compounds are formed. In the disordered amorphous regions of metals, the cohesion is due to Van der Waals

forces of high magnitude. This is merely another way of stating that the attractive force does not reach the magnitude of a valence bond. When metals are cold drawn, however, there is a much greater tendency toward crystalline lattice formation with an accompanying increase in cohesive strength.

b. Glass.- Silicate glasses are the most common and have been shown by Warren (reference 12) to consist of a random network of silicon and oxygen atoms with each silicon atom tetrahedrally bonded to four oxygen atoms (fig. 2). All the oxygen atoms, however, are not bonded to silicon atoms; thus interstices of varying sizes exist throughout the structure. When the composition of the glass is modified by alkali or alkaline earth metals, these interstices become more numerous and each metal ion is surrounded by oxygen atoms with unsatisfied charges and is consequently linked to these through coordinate covalent attractive forces.

Glass structures, in general, are composed of oxides such as SiO_2, B_2O_3, P_2O_5, Al_2O_3, and so forth, which are known as network formers and oxides of alkali or alkaline earth metals such as Na_2O and CaO, which are known as network modifiers.

c. Wood.- The structure of wood is, as is true of most complex natural materials, very imperfectly known. Predominantly, it consists of long fibers of cellulose joined together by means of lignin. The structure of cellulose is that of a chain composed of glucoside units linked to one another through C-O-C bonds. Neighboring chains are intramolecularly held together by means of hydrogen bonds.



Lignin is not a single substance; its chemical nature varies from species to species.

The bond between cellulose and lignin is in itself a problem of adhesion. Although the greatest strength of wood lies in the direction of the cellulose fiber, many of the properties of a given wood depend on the nature and amount of its lignin component.

d. Natural and synthetic high polymers.- High polymeric materials include all substances which may be considered as consisting of large multiples of monomeric units, formed either by polymerization or condensation. Cellulose, rubber, and vinyl resins are examples of polymeric materials. There are two types of high polymeric compounds:

Thermoplastic - two-dimensional molecules

Thermosetting - three-dimensional molecules

These two classes of materials have arrangements somewhat analogous to those of asbestos and diamond, respectively.

1. Thermoplastic polymers (reference 13).- If the attractive forces between chains are strong and the fine structure permits of easy lattice formation, a high degree of crystallinity and hence cohesion will result. Such materials fall in the fibrous area of figure 3. In fibers such as cellulose the individual chains are probably held together by means of hydrogen bonds through the OH groups. Other fibers of high strength, such as nylon, are probably joined by similar forces.

The forces between molecules of polystyrene, polymethyl methacrylate, and related materials are not as great as those between cellulose and nylon molecules. These materials fall in the plastic area of figure 3. There are no very reactive centers, such as hydroxyl or amido groups for forming hydrogen bridges between chains. The most likely assumption to explain their strength in films, filaments, and so forth, is that they are held together by Van der Waals forces which are strong because of the great asymmetry of the monomer molecules. The vinyl polymers are not as asymmetric as polystyrene or methyl methacrylate and they tend toward rubbery characteristics.

Rubbery materials owe their properties to very much weaker Van der Waals forces between the molecular chains. A tensile load applied to a rubber causes

an actual change in the configurations of the chains; the coiled equilibrium state is changed to the stressed chain state. These materials are located in the rubbery area of figure 3.

2. Thermosetting polymers.- Cured thermosetting resinous materials, such as phenol-formaldehyde and urea-formaldehyde condensates, are composed of a three-dimensional cross-linked network, analagous to the diamond structure.

In order for such a structure to be formed, it is necessary that at least one of the reactants in the polymerization mixture have three or more active centers at which bond formation can occur. In phenol-formaldehyde resin, for example, the benzene ring is capable of forming bonds at the para and two ortho positions with respect to the hydroxyl group. The cohesive forces in such a material are very strong covalent bonds, and consequently, the gross physical properties are those of great strength and stability.

C. THE NATURE OF ADHESION

1. Theories Concerning Adhesion

The investigation into the nature of adhesion has as its objective the establishment of the most desirable physical and chemical properties for an adhesive between two given surfaces so that maximum structural strength can be obtained. In the past two decades considerable interest has been aroused in this subject. Probably the greatest effort in this direction was that undertaken in Great Britain, the results of which are described in the three reports of the Adhesives Research Committee under the auspices of the Department of Scientific and Industrial Research. The greater part of this investigation was conducted by J. W. McBain and coworkers (reference 14). At the same time, work was being done in this country at the U. S. Forest Products Laboratory by F. L. Browne and T. R. Truax. Both organizations were interested in the mechanism whereby two surfaces of an adhesively formed joint are held by the adhesive.

McBain as a result of his investigations arrived at the conclusion that two types of bonded joints may be formed:

Mechanical - between porous surfaces

Specific - between smooth, dense surfaces

Browne and Truax (see references 15 and 16), however, maintained that although some of the strength of the bond between two porous surfaces must be attributed to tendrils of adhesive which enter the pores, this mechanical interpenetration cannot account for more than a small fraction of the joint strength.

a. McBain's experimental work.- McBain drew his conclusions from the following experiments:

1. McBain prepared joints between surfaces of silver to silver and silver to wood. He varied the character of the silver surface by welding silver gauze at several points in single and double layers to the smooth surface and obtained qualitative results for the strength of bonds prepared with a proprietary gelatin glue:

Joint[1]	Strength	Remarks
1. Ag:glue:Ag	0	Glue film moist.
2. $(Ag)_g$:glue:$(Ag)_g$	0	Glue film moist.
3. Ag:glue:wood	0	Glue film only on wood when joint was broken.
4. $(Ag)_g$:glue:wood	Weak	Stronger than 3; glue film partly on silver gauze and partly on wood.
5. $(Ag)_{gg}$:glue:wood	Moderate	Stronger than 3 or 4; glue film partly on silver gauze and partly on wood.

[1] $(Ag)_g$ = Silver to which was welded one layer of gauze.

$(Ag)_{gg}$ = Silver to which was welded two layers of gauze.

Similar results were obtained using gum arabic and molten shellac, respectively, as adhesives.

2. Surfaces of hard and soft woods were treated with three successive coatings of ebony stain before bonding and the joint strengths were compared with those of unstained surfaces by breaking manually. In the case of soft woods the joints prepared with either the stained or the unstained surfaces could not be broken by hand, but in the case of the hardwoods, the stained joints broke. McBain concluded that the stain was able to fill the pores of the hardwoods, but not of the soft woods, thus preventing mechanical penetration by the glue of the hardwoods, which consequently gave weak joints.

3. If adhesion to porous surfaces is mechanical, the joint strength must be directly related to the tensile strength of the glue. McBain, using the method of Farrow and Swan (reference 17), prepared adhesive films by casting on thinly greased ferrotype plates. Films and specimens of bonded walnut wood prepared with a sodium silicate glue and an animal glue, with and without the addition of dextrose, respectively, had the following strengths:

Glue	Strength (psi)		
	Glue film in tension	Walnut joints	
		Shear	Tension
1. Animal glue	12,000	1500	825
2. Animal glue mixed with an equal part of dextrose	Less than 1000	600	420
3. Sodium silicate	600	700	---

4. Specific adhesion requires wetting of the surface not only by the solvent, but also by the glue. McBain attempted to show that gelatin does not wet several porous surfaces with which it forms strong joints. (See references 18 and 19.) To do this, he measured the water absorption of filter paper, viscose rayon, and fused silica, and then measured the change in concentration brought about by the addition of these materials to aqueous solutions of glue. He found that only with fused silica did the concentration become lowered. This he interpreted as showing that water was being removed from the solution by porous substances such as filter paper and viscose rayon, but no glue was being adsorbed.

5. McBain believed that if specific adhesion is operative, the attractive field of the solid surface should impose

an orienting influence upon the molecules of the very thin adhesive layer. In such a case the glue structure should differ in a glue line from that of a solid block of glue. Studies of X-ray patterns from glue films on wood revealed no such difference.

b. Forest Products Laboratory experimental work.- Browne, Truax, and other workers gave the following interpretation to these experiments:

1. Some degree of specific adhesion is a necessary prerequisite of mechanical adhesion in the case of materials having such a fine degree of porosity as wood. Red oak surfaces, selected because of the large pores, were joined with Lipowitz metal alloy (reference 16) which has a melting point of 60° C, does not wet wood, and has a viscosity not unlike that of woodworking glues. The joint was easily separated by hand and the film was removed intact.

Glue solutions contain considerable volumes of water or other solvents. As the joint dries, the glue contracts throughout its entire mass. If the interpenetrating tendrils did not adhere tenaciously to the cell walls, they would shrivel and the entire interface between wood and adhesive would disintegrate. Photomicrographs of glued sections showed that in all cases the glue had remained attached to the cell walls and in drying had contracted from the center of each tendril, leaving a hollow cylinder.

2. In the staining of hard and soft woods prior to gluing, the pine probably absorbed the oil stain more readily than did the mahogany; thus enough of the surface of the pine was unaffected to form a good joint. If sandpaper were used to clean the stained mahogany surfaces the filled pores would be unaffected; whereas cells of the wood would be exposed on the surface to be bonded. McBain's experiment was repeated using paraffin wax and collodion, respectively, to seal the pores and the surfaces were sanded before gluing. Good joints were obtained although penetration was greatly reduced as evidenced by photomicrographs.

3. McBain's relationship between the tensile strength of glue films and the shear and tensile strength values of glue joints was based on an experiment in which gluing methods assuming mechanical adhesion were used. Thus, the glue was prepared in a thin solution, applied hot to heated wood surfaces and pressed immediately in order to fill the wood as much as possible with glue.

This experiment was repeated on several species of wood glued with sodium silicate, animal glue, and animal glue containing two concentrations of dextrose. The tensile strength of walnut joints, and the shear strength of maple joints glued with these substances is as follows:

Glue	Strength (psi)		
	Glue film in tension[a]	Wood joints	
		Shear[b]	Tension[c]
1. Animal glue	12,000	3950	1400
2. Animal glue-dextrose mixed (1:1)	Less than 1000	3350	1300
3. Animal glue-dextrose mixed (1:1.5)	----	3150	----
4. Sodium silicate	600	3000	950

[a]Values determined by McBain

[b]Maple block shear

[c]Walnut joints

In each case the glue was applied in concentrated solution to one surface and allowed to dry until the glue line retained a finger print and then assembled. The joints so obtained were much stronger than those of McBain. Although there is some loss in joint strength when the glue is weakened, this loss is not so great as indicated by McBain and, furthermore, the greater number of failures in these joints occurred in the body of the glue and not at the interface between the glue and the wood, so that the failure was not one of adhesion.

4. McBain's conclusion regarding the lack of adsorption of gelatin from aqueous solution is not necessarily correct. If water is more attracted to wood than is the gelatin, it will be selectively adsorbed from solution, thus giving the appearance of negative adsorption for gelatin.

Campbell (reference 20) has recently advanced the theory that glue is adsorbed to wood surfaces through a monomolecular layer of water which is directly attached to groups on both the wood and glue surfaces.

5. Although the Forest Products Laboratory workers did not comment on McBain's fifth experiment, later investigators studying the effect of surfaces on lubricant films have found that attractive forces between a surface and an adsorbed layer do not operate through much more than a monomolecular layer of the liquid. (See reference 21.) This is in agreement with the findings of Lennard-Jones (reference 8) who calculates the electrostatic attraction between surfaces and charged particles. In such a disorganized macroscopic system as an animal glue film it is not likely that orientation effects present in a monomolecular layer could be detected by the method used by McBain.

2. The Chemical Properties of Surfaces (reference 10)

It has been shown that adhesion to any porous surface is not primarily a mechanical phenomenon. Evidence is accumulating in every pertinent field that molecular attractive forces are responsible for the bond between any two surfaces. A knowledge of the chemical nature of surfaces is necessary in order to establish the type of bond formation which may occur.

The surfaces of solids are entirely different from their internal structures. The simple treatments of grinding and polishing differ very greatly in their effects on surface nature. In metals, especially, X-ray diffraction patterns show that grinding removes sections of the surface without appreciable distortion of the remaining crystal structure; whereas polishing removes the promontories and deposits materials from them in the crevasses, leaving a smooth transparent amorphous film known as the Beilby layer. This layer has more of the characteristics of a liquid than a solid and is much more reactive. If, for example, a metal vapor is condensed upon a polished metal surface, a crystal pattern is first obtained, but on standing this changes to that of a completely amorphous structure. If a nail is hammered into a crystalline surface, the amorphous ring around the hole will be more readily attacked by acids than the remainder of the surface. Glass surfaces when polished are rendered doubly refracting to a depth of 3000 angstrom units.

Solid surfaces are very irregular in their constitution. Thus, the surface of even a monocrystalline metal will contain different faces, edges, corners, and projections. For this reason, all parts of a given surface will not have the same catalyzing ability and consequently the same degree of

affinity for adhesive substances. Experiments show that different faces of the same crystal exert very different forms of attraction upon dyes. Thus, if lead nitrate is immersed in a solution containing methylene blue and picric acid, some of the surfaces will be blue and some yellow; neither film is contaminated with traces of the other dye.

The greater the activity a surface possesses, the more likely it is to be contaminated with impurities, which are present in the air. Freshly split mica surfaces will sieze each other tenaciously, but if they are freely exposed to circulating air currents they lose this activity after a few minutes. It has been demonstrated that if these surfaces are covered, they will retain their attraction for each other for several days. Accidental traces of grease will greatly reduce the wetting properties of such surfaces and even slight contamination will increase the contact angle with a liquid.

The purification of a surface is, consequently, of the greatest importance if strong tenacious glue lines are to be obtained. This is by no means a simple operation. It is possible to clean the surface of glass by heating with a flame for a short time, provided the contamination is not excessive, but this treatment cannot be applied to most other surfaces as they would become sintered. Metals may be cleaned with benzene or by slight etching with acids followed by washing with distilled water and drying with alcohol and ether. It has been demonstrated experimentally at the Curtiss-Wright Laboratories that sanding of wood surfaces with the finest grade of sandpaper immediately before gluing gives a much better bond than that obtained with glazed surfaces produced by planing or with roughly sanded ones; thus the sanding operation is one of cleansing.

3. The Mechanism of Adsorption

a. Adsorption phenomena, general.- Determination of the heats of adsorption and chemical investigation of adsorbed films on solid surfaces show that two distinct types of forces are involved. When a gas is attracted to a surface by means of Van der Waals forces, the heat of adsorption is 4000 calories per mole or less, and the variation of a few degrees in temperature results in a simple pumping off of part of the film. If, on the other hand, an actual covalent bond is formed, as is the case with oxygen adsorbed on carbon, the heat of adsorption will be of the order of 10,000 calories per mole and may even reach as high a value as 200,000 calories per mole. In this latter case, the temperature required

to release the adsorbed film is sufficient to pyrolyze the chemical bond. Very frequently this bond is stronger than the cohesive bonds of the adsorbing material and the compound formed between it and the gas is released as such; thus, when oxygen is adsorbed on charcoal, heating results in the liberation of carbon monoxide and carbon dioxide.

Depending on the temperature, the same gas may be adsorbed by Van der Waals forces or by primary chemical forces. Polanyi (reference 22) pointed out that the free valences of a surface would not attract the impinging molecules of a gas until these had surmounted a potential barrier. In other words, only those molecules which have a certain energy value will combine chemically with the surface. This relationship is well illustrated by the behavior of hydrogen on nickel at various temperatures between -200° and 100° C at different pressures. Thus, at 600 millimeter pressure the curve of volume adsorbed plotted against temperature is given in figure 4. (See reference 23.) At -190° to -180° C adsorption takes place almost instantaneously through Van der Waals forces. The amount of gas adsorbed decreased to a minimum at about -160° C because of the increase in kinetic energy of the gas molecules. Above -160° C primary bond formation begins to occur. Adsorption reaches a constant value between -110° and 0° C. Further increase in temperature results in the pyrolysis of the chemical bonds.

b. Adsorption phenomena at glue lines.- It is evident that the primary consideration in selecting a suitable adhesive for a given surface is an evaluation of the relationship between active groups on the molecules of the adhesive and the surface. At the present time, the difficulties involved in measuring the exact area of a solid surface and the lack of knowledge with respect to the configurations, molecular weights, and kinetic behavior of high polymeric substances make it impossible to define mathematically the total bond energy at a glue line interface. It should be possible, however, to construct a theoretical picture of this region which would lead to a better understanding of the phenomenon of gluing.

1. Metal surfaces

(a) Metals to metals (direct).- The art of metal bonding depends very greatly on the uses of solders and welds. The chemistry of alloy formation furnishes considerable information on the nature of the attractive forces involved. If two metals are very closely related - for example, copper

and gold atoms of the one will replace atoms of the other in a crystal. When there are certain definite proportions - for example, CuAu and Cu_3Au, the distribution in the crystal will resemble that of an ionic compound such as NaCl. If the metals are dissimilar they may actually form chemical compounds which have their own crystal structure and have chemical and physical properties which are quite different from those of the constituent elements. Thus, the melting points of sodium and mercury are 97.5° and -38.7° C, respectively; whereas the substance Hg_2Na melts at 360° C.

Although the exact relationships which hold in the crystalline state are not exactly those obtained in a soldered or welded joint, the same general laws apply. Thus, the experience with solders has been that the strongest and most permanent bonds are formed between metals having optimum chemical affinity. (See reference 24.) In all cases where the metal surface remains solid, a good bond with the liquid solder is best obtained when a wetting agent or flux is employed to cleanse the interface.

(b) Metals to enamels.- When metals are glazed with vitreous enamels, it has been observed that the ground coat between the metal and the glaze must contain cobalt or a similar oxide. The role of the cobalt compound has been the subject of much controversy, but a plausible theory is that it serves as a catalyst in forming the bonds between atoms of the metal and oxygen atoms which are attached to silicon in the enamel. The interfacial bond between a vitreous and a metallic surface, according to Weyl (reference 25) must be of the chemical type in order to explain the stability of such a system. King (reference 26) has reported that in the case of good bonding between iron and enamels, atoms of iron partially penetrate into the enamel in the form of dendrites which remain attached to the iron surface.

(c) Metals to rubber.- It has long been known that extremely good bonds are obtained by vulcanizing rubber onto certain metallic surfaces. Evidence shows that the rubber is actually attached to the surface through a sulfur bridge. The bond is obtained more easily with brass than with iron (reference 27), a fact which correlates with the greater ease of the sulfide formation of copper and zinc than of iron.

(d) Metals to resinous adhesives.- An example of primary bond formation between a resin and a metal surface has been described by Doolittle and Powell. (See reference 28.) These workers developed a vinyl chloride-acetate copolymer resin VMCH for use as a metal lacquer by polymerizing

a small amount of maleic acid with resin, thus giving free carboxyl groups on the chain ends of the polymer. The amount of acid must be controlled to a small proportion, because 5 percent or over causes excessive corrosion of the metal. As low as 0.1 percent, however, gives improved adhesion and the optimum amounts are between 0.3 to 1.0 percent. That the carboxyl group is responsible for adhesion was proved by forming the sodium salt of the resin, thereby destroying its adhesive properties. Diesters of the acid showed poor adhesion, but monoesters were almost as effective as the acid itself. This development is an excellent example of what may be expected from a careful study of the chemical nature of adhesion.

In his work for the Adhesives Committee, McBain investigated the adhesion of a large number of substances to metals. He found a close relationship between the atomic volume of the metal and the bond strength of a given adhesive. With shellac, for example, there is almost a linear relationship between atomic volume and tensile strength of the joint, as shown by the following data:

Metal	Atomic volume	Tensile strength of shellac-glued joint (psi)
Nickel	6.7	3500
Copper	7.1	3300
Aluminum	10.1	2800
Tin	16.2	1100
Lead	18.2	600

Investigators at the Resinous Products and Chemical Company have found that Redux joints are stronger with high valency metals than with low, and that with different grades of steel the adhesion is poorest with those which oxidize readily, such as silicon steel.

2. Wood surfaces

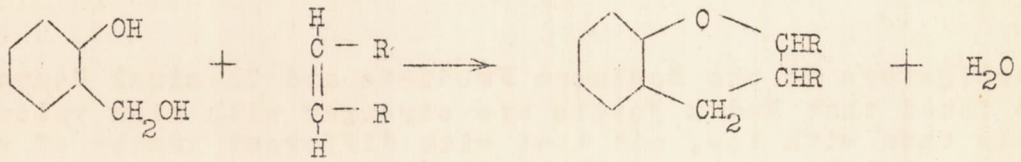
(a) Wood to adhesives.- Adhesives for wood have been more thoroughly investigated than for any other material. The mechanism for the bond between wood and phenolic resins, which is much stronger than the cohesive bonds of wood, is very probably hydrogen bridging between hydroxyl groups in the two materials. The reactivity of the phenolic hydroxyl groups is indicated by the fact that water-insoluble phenol-formaldehyde resin will dissolve readily in sodium hydroxide

solutions of moderate concentration and even the cured resin is attacked by strong alkaline solutions.

The probable structure of urea-formaldehyde resins indicates that much of their bonding power resides in the amido ($-NH_2$) and imido ($=NH$) groups which are present in large numbers in the uncured state. These groups will also participate in hydrogen bonding to other amido, imido, or hydroxyl groups. Campbell (reference 20) postulates that, instead of direct bonding to cellulosic hydroxyls, a molecule of water serves as an intermediary bonding material between wood and urea resins. The rapid failure of such structures when subjected to alternate wetting and drying was regarded as evidence of such bonding. Introduction of high-boiling water-insoluble alcohols into the adhesive mixture improved the resistance of the bond to delamination under the cycle conditions.

(b) Wood and metal to adhesives.- Most adhesives which form satisfactory bonds to metals are not suitable for bonding wood and vice versa. Several adhesives have been developed recently, however, which consist of combinations of rubbers with phenolic resins. The bonding is accomplished by heat, which cures the phenolic resin in a strong bond to the wood and the rubber to the metal.

It has been found by Cunneen, Farmer, and Koch (reference 29) that olefins form compounds with saligenin, which is the precursor of phenol-formaldehyde resin.



(Saligenin) (Olefin)

It has also been observed that an oxidized rubber reacts readily with maleic acid, phenol, and formaldehyde to give a modified rubber-phenolic resin. (See reference 30.)

A reaction between rubber and a curing phenolic resin probably occurs in this same manner, the terminal salegnin grouping of the resin attaching to the double bond in the surface molecules of the rubber. Thus a primary chemical bond would be formed, accounting for the high shear strength observed for these rubber-resin adhesives.

3. Glass surfaces.- The effect of cobalt oxide in bonding vitreous enamels to metal has already been mentioned. Stannous chloride is used to promote adhesion in plating silver and other metallic films on glass. The strength of the bond between sodium silicate and glass indicates that primary chemical bonds are involved.

4. Rubber surfaces.- Strong bonds may be obtained between rubber surfaces through Van der Waals forces as well as through chemical bonds. In the first case, a solution of rubber applied to a rubber surface leaves a fused structure which has good bond strength. In the second case, vulcanization proceeds across the interface of two rubber surfaces in the same manner as within the two original masses. This is evidenced by the bond strength of vulcanized patches on tires.

5. Plastic surfaces.- It has been found by Barkhuff and Carswell (reference 31) that a thermosetting resin cured at one temperature gives evidence of further cure at a higher temperature. This indicates that such a resin may frequently retain a degree of reactivity which would be sufficient for chemical reaction at the surface with a curing thermosetting adhesive. In the bonding of any resinous surface cured in a mold or between press platens, it is very important to remove any waxes used in their fabrication from the surface in order to permit adsorption of the adhesive material.

Thermoplastic materials are usually swelled or dissolved by organic solvents. Consequently, they may be bonded by the addition of a solvent which attacks both surfaces and allows an interdiffusion of the two materials. This permits the Van der Waals attractive forces to exert a maximum effect brought about through hydrogen bridges.

4. Fundamental Approaches to the Determination of the Nature of Adhesion

a. The Bartell cell (reference 32).- It has long been known that the affinity of a solid substance for a liquid was directly related to the shape of a drop of the liquid on a smooth surface of the solid. By measuring the angle of contact, it is possible to calculate the adhesion tension at the interface:

$$A_{1,2} = S_2 (\cos \theta) \quad \text{where} \quad \theta > 0 \qquad (1)$$

where

A adhesion tension

S surface tension

θ contact angle

Subscripts

1 solid

2 liquid

This device is useful where the contact angle is finite, but for the purpose of a study of adhesives it is much more important to measure the attraction of a solid for liquids which form a contact angle of zero. In other words, the best adhesion is given by substances which have a greater adhesive attraction for the surface than cohesive attraction within themselves.

Bartell and coworkers have found that by using two immiscible liquids, one which wets the surface very well and one which wets it very poorly, it is possible to measure adhesion tension if several other values are known or can be calculated. This is based on the use of a displacement cell now known as the Bartell cell, which is shown in figure 5.

The solid substance is powdered to a uniform particle size carefully cleaned and dried and packed into the cell compartment by a special technique, such that two-thirds of the plug is saturated with the poorly wetting liquid and one-third with the liquid which is to be measured. The spaces between the powdered particles behave like minute capillary pores, the average radius of which may be determined by means of the formula for the rise of liquids in capillary tubes as well as by Poiseuille's formula for the rate of flow of liquids through capillary tubes. Values calculated by both of these methods check the pore size with a high degree of accuracy.

After packing the cell, the equilibrium pressure required to displace the poorly wetting liquid by the highly wetting liquid is measured. By means of the following formulas, the adhesion tension is calculated:

$$\cos \theta_{2,3} = \frac{981 \text{ Pr}}{2 S_{2,3}} \qquad (2)$$

$$A_{1,2} - A_{1,3} = \cos \theta_{2,3} \; S_{2,3} \tag{3}$$

$$A_{1,3} = \cos \theta_{1,3} \; S_3 \tag{4}$$

$$A_{1,2} = \cos \theta_{2,3} \; S_{2,3} + A_{1,3} \tag{5}$$

where

A adhesion tension

P displacing pressure

S surface tension

θ contact angle

r average pore radius

Subscripts

1 solid

2 highly wetting liquid

3 poorly wetting liquid

An example of such a calculation is given by solid carbon, carbon tetrachloride, and water (carbon = 1; carbon tetrachloride = liquid 2; water = liquid 3):

$\theta_{1,2} = 0^\circ$ $\qquad$ $r = 9.3 \times 10^{-6}$ cm

$\theta_{1,3} = 40^\circ\ 35'$ $\qquad$ $P_{2,3} = 6935$ g/cm^2

$S_{2,3} = 44.54$ dynes/cm $\qquad$ $S_3 = 72.08$ dynes/cm

$$\cos \theta_{2,3} = \frac{6935 \times 981 \times 9.3 \times 10^{-6}}{2 \times 44.54} = 0.7103$$

$$A_{1,2} - A_{1,3} = (0.7103)(44.54) = 31.63 \text{ dynes/cm}$$

Adhesion tension water:carbon = $A_{1,3} = (\cos 40^\circ\ 35')(72.08)$
$= 54.74$ dynes/cm

Adhesion tension CCl_4:carbon = $A_{1,2} = 54.74 + 31.63 = 86.37$ dynes/cm

A possible means for attacking the problem of adhesion of high polymers to solid surfaces would be the measurement of adhesion tension values of polymers of low molecular weight. Thus, monomeric vinyl acetate and pure fractions of several polyvinyl acetates of low molecular weight may establish a useful relationship between adhesiveness and polymer size.

b. Heat of wetting (reference 33).- When a solid is immersed in a liquid it may or may not be dissolved, but in either event a heat change occurs. The heat liberated in the latter case is known as the heat of immersion or wetting and is directly related to the energy of attraction between the two substances. Harkins and coworkers have devised a calorimetric method for measuring this heat and the results have been applied in the paint and varnish industry in selecting suitable vehicles for pigment particles.

c. Heat of formation.- In the investigation of the nature of adhesion, the heats of formation of chelate compounds as well as ion exchange reactions of certain groups such as hydroxyl and carbonyl should throw some light on the strength of bonds involving such groups. Phenolic resins remove anions from water by exchanging hydrogen for the anion on the hydroxyl group. This takes place readily with sodium and should bear some relation to the heat of reaction of sodium phenolate, resorcinate, and so forth. Although unmodified phenolic resins are poor adhesives for metals, because of incompatible physical properties, they do form such strong bonds to steel molds that the removal of a molded object may be effected only when waxes are used to prevent bond formation.

II. PROPERTIES OF ADHESIVELY BONDED STRUCTURES

A. PHYSICAL STRENGTH OF BONDS

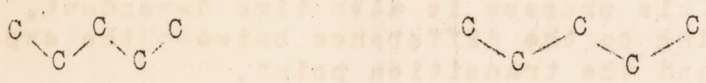
1. Mechanical Behavior Phenomena

It has been previously shown that high polymers consist of a weblike distribution of molecules which, in addition to being mechanically intertwined, are attracted to one another by forces ranging from weak Van der Waals forces to strong chemical bonds. Any high polymer at a given temperature will, consequently, be either rigid or soft, depending on the magnitude of these bonds. There is a definite temperature for each

substance below which it is appreciably more rigid than it is above that temperature. This point is known as the transition point and is a characteristic of materials bonded by Van der Waals forces.

When a high polymer is placed under a given stress it will suffer a deformation, the extent of which depends on the temperature and the amount and rate of loading. The deformation may be resolved into three components. (See reference 34.)

a. Ordinary elastic deformation (d_{OE}).- The individual links in the chain molecule may be stretched by altering the bond angle under an applied load.



d_{OE}

The forces required to bring about such a strain are very high, being of the order of Young's modulus for metals. Thus may be defined a modulus of elasticity (G_{OE}) for this process which has a magnitude of 10^4 to 10^6 kilograms per square centimeter. Such a deformation will appear or vanish immediately as the load is applied or released. It is completely independent of temperature.

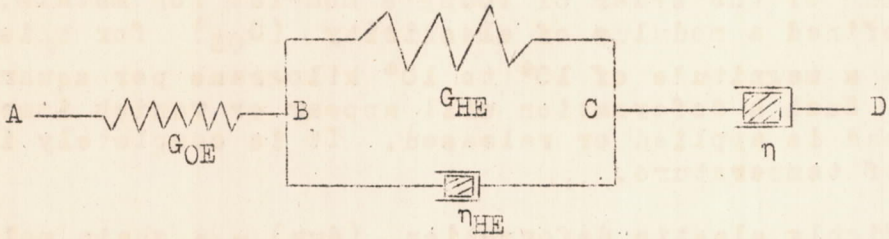
b. Highly elastic deformation (d_{HE}).- A chain molecule has considerable length with respect to molecular dimensions and the single covalent bonds do not prevent free rotation of the atoms in the chain. Consequently, the maximum possible distance between ends of a carbon chain is never reached in equilibrium. It seems most likely that the chain is more or less coiled along its entire length and the distance between ends will have a definite range for a given kinetic energy of the molecule. In order to increase the distance between the ends of these molecules, it is necessary that a distorting load be sufficient to uncoil the chain. In addition to the energy required for uncoiling, some energy must be expended in overcoming the attractions between points along the chain and similar points on neighboring molecules. This latter effect accounts for the fact that this process is temperature dependent.

The modulus of elasticity (G_{HE}) for a material which may be distorted by this process is of the order of 10 to 100

kilograms per square centimeter, and very great distortions are obtained; rubber, for example, may be extended 1000 percent. Once the load is removed the coiling process will begin, but requires time to reach equilibrium. This equilibrium is attained by simple revolution of the atoms about the single bonds and by overcoming attractive neighboring forces which retard the process.

c. Viscous deformation (d_{visc}).- Above the transition temperature a thermoplastic material will be principally distorted by separation of the molecular chains. This means that the kinetic energy of chain motion is sufficient to overcome the Van der Waals side forces, thus allowing the molecules to be drawn along each other without a greatly retarding friction. This process is also time dependent, the rate of flow depending on the difference between the experimental temperature and the transition point.

d. Deformation equation.- The total distortion of a material exhibiting all these characteristics may be represented by the following diagram:



Each element in the diagram will be displaced to an extent dependent on the load. The increase in distance between A and D under a given load is the total distortion for that load. Thus, there may be written:

d_{OE} increase in distance AB

d_{HE} increase in distance BC

d_{visc} increase in distance CD

This process has been represented mathematically by Alexandrov and Lazurkin (reference 35) and modified by Simha (reference (36) as follows:

$$D(t) = d_{OE} + d_{HE}(\infty)\left[1 - \exp\left(-\frac{t}{\lambda}\right) + \frac{t\tau}{\eta}\right] \qquad (1)$$

The total deformation (D) is expressed as a function of rate of loading, and as the sum of the three types of deformation already discussed. The significance of each of these elements is as follows:

1. Ordinary elasticity

$$d_{OE} = \text{ordinary elastic deformation}$$

$$= \frac{\tau}{G_{OE}}$$

where

G_{OE} ordinary shear, tension, or compression modulus of elasticity

τ stress

2. High elasticity

$$d_{HE}(\infty) = \text{ultimate high elastic deformation}$$

$$= \frac{\tau}{G_{HE}}$$

where

G_{HE} modulus of high elasticity

This factor is modified by the exponential term which expresses the dependency on rate of loading and the viscous element:

t time elapsed between start of loading and the instant of observation, rate of loading being constant

$$\lambda = \frac{\eta_{HE}}{G_{HE}}$$

η_{HE} the viscous effect associated with the highly elastic deformation

3. Viscosity.- The final term expresses the deformation brought about by the viscous flow of the material modified by the rate of loading.

This equation for the deformation of high polymers is only a first approximation. Other expressions have been developed, based on thermodynamics, which involve considerations of crystallization phenomena. It is expected that further developments in the interpretation of mechanical behavior of high polymers will materially contribute to a better understanding of glue line phenomena.

2. Effect of Thickness of Glue Line on Strength

a. Experimental evidence.- An investigation of several adhesives for wood by Poletika (reference 37) reveals that joint strength is inversely proportional to the thickness of the glue line.

Glue layer thickness (in.)	Number of samples	Average shear strength (psi)	Average wood failure (percent)
0.002	5	1500	37
.003	5	1330	40
.004	11	1450	25
.005	17	1410	22
.006	6	1340	21
.007	7	1150	29
.008	8	1100	19
.009	10	1180	11
.010	9	1200	9
.011	6	900	12
.012	7	940	11
.013	7	920	6
.014	4	840	4
.015	5	850	4
.016	3	670	5
.017	4	790	3
.018	4	480	2
.019	2	560	5
.020	1	520	3

However, starved joint formation will occur if too little adhesive is applied to porous surfaces and would result in a weak joint because of lack of film continuity.

Similar relationships were found by Crow (reference 38) in a study of soft soldered joints. He found that, by making the film very thin, joint strengths as high as 11 tons per square inch were obtainable. This value is several times the tensile strength of the solder.

b. Probability of flaws.- The relationship between average strength and rod or fiber length has been discussed by Peirce (reference 40) for cotton, Bellinson (reference 41) for rayon, and Anderegg (reference 42) for glass. If a fiber 10 inches in length is broken, the rupture will occur at the weakest spot. If another fiber exactly like the first is divided into ten 1-inch lengths and each of these is broken, a range of breaking loads will be obtained, the lowest being equal to the one obtained on the first fiber. Thus, the shorter the specimen the less will be the probability that it will contain the weakest spot. This same analogy can be applied to glued joints; the average strength of 10 thin ones should be greater than that of one ten times as thick.

To test this assumption, Bikerman (reference 39) prepared joints between brass blocks and steel cylinders with paraffin wax. He controlled the thickness of the wax film in each joint so that 100 joints were obtained having thicknesses of approximately 57 μ and 40 of approximately 540 μ. The operational variables were minimized by preparing 10 thin joints and 4 thick joints in 10 successive groups. The tensile load was applied by suspending gram weights from the samples. The results of these tests are as follows:

Strength of Paraffin Wax Joints
(kg/cm^2)

Group of specimens	57 μ			540μ
	Maximum	Mean	Minimum	Mean
1	35.0	27.5	18.0	17.5
2	35.0	29.5	20.0	15.5
3	32.0	26.0	20.5	16.5
4	31.0	25.0	18.5	14.5
5	25.5	22.0	17.0	13.0
6	32.0	24.5	19.5	18.0
7	28.0	22.5	14.5	13.0
8	29.5	23.5	20.5	13.0
9	28.5	23.0	16.5	14.5
10	29.5	22.5	17.0	13.0
Average	30.6	24.6	18.20	14.85

The average value obtained for the thin joints compares with the value of 18 kilograms per square centimeter for paraffin to brass obtained by McBain and Lee. (See reference 14.) The tensile strength of paraffin measured by Konstantinova (reference 43) is 7.45 kilograms per square centimeter and for joints thinner than 0.01 millimeter, 14.8 kilograms per square centimeter. McBain and Lee obtained the following values for varying thicknesses of shellac joints; these show similar relationships to Bikerman's values:

Joint	Strength of Joints (kg/cm^2) Thin 57 μ	Thick 540 μ	Ratio thin/thick
Al:shellac:Al	145.0	75.0	1.93
Ni:shellac:Al	190.0	110.0	1.73
Brass:paraffin:steel	24.6	14.8	1.67

Bikerman analyzed his data by means of a simple application of probability theory as follows:

Let m = number of specimens of length l
n = number of specimens of length l/n

If it is assumed that the probability of finding a weak spot is a function of length, one in n of the thin joints will contain the weak spot which would have caused the failure of the thick joint. The tensile strength of this joint would accordingly be identical with that of the thick joint. If this reasoning is correct, the low values in each batch of the paraffin joints of 57 μ thickness should not differ significantly from the average values of those of 540 μ thickness.

The difference between the low values of the thin joints and the average values of the thick joints was found to be significant, but the probability theory accounts for approximately two-thirds of the difference in strength. This is shown as follows:

Let m = number of values
t = a measure of the significance of the difference
$x_1, x_2, x_3 \ldots x_{10}$ = the differences in the same group between the minimum values of the thin joints and the average values of the thick joints

and x = the difference between the total averages for the minimum values of the thin joints and the average values of the thick joints

Then $m = 10$

$x = 18.20 - 14.85 = 3.35$

and $t = x\sqrt{m(m-1)}\Big/\left[(x_1-x)^2 + (x_2-x)^2 + \ldots + (x_{10}-x)^2\right]^{\frac{1}{2}}$

$= 5.1$

Since, when $m = 10$, only one value of t in 100 will exceed 3.25 by chance, the difference between the low values for the thin joints and the average values of the thick joints is significant. The discrepancy between these values is explained by Bikerman as due to crystallization differences in the thick and thin sections of wax.

c. Surface smoothness.- In order to obtain a strong joint, a smooth surface is more desirable than a roughened one, inasmuch as the depressions in the roughened one must be filled in addition to the gap between the two surfaces. Furthermore, if the surface is deeply scored, there is always a possibility that air bubbles will be trapped in the glue line. The existence of such points of interfacial discontinuity will cause high concentrations of stresses in their vicinity which will result in premature failure of the entire bond when external loads are applied.

d. Effect of curing conditions.- When a thermosetting resin adhesive is used, it is cured in the joint with pressure and very frequently with heat. Thus, it reaches an equilibrium state under an abonrmal set of conditions. When the pressure and heat are released, the resin has a tendency to reach an equilibrium at a lower pressure and temperature, but is restrained by its bond to two surfaces. This results in a stress at the glue line. If the glue line is very thin, these stresses will be very small with respect to the bond strength and the glued joint will be able to support much higher loads. Similar stresses are set up in adhesive films which are formed by evaporation of solvent.

3. Problem of Thermal Expansion Differences

Inorganic materials such as glass and metals are characterized by relatively low coefficients of thermal expansion; whereas organic materials in general have high thermal expansion coefficients. Hence, stresses are developed in joints

prepared with these two types of materials when the temperature differs appreciably from that at which the bond was formed. If the molecular cohesion of one of the materials is sufficiently low, the stresses developed at the glue line can be relieved by flow. However, such materials usually undergo excessive flow at temperatures above the softening point and have poor bond strengths. For materials which have high molecular cohesion and hence undergo very little flow, such as the thermosetting resins or most materials at low temperatures, the stresses developed because of different thermal expansion coefficients are not readily relieved by molecular flow.

For the latter type of material it is necessary to add an ingredient to the adhesive to adjust its thermal expansion coefficient to approximately that of the adherend. Investigations at the National Bureau of Standards (reference 44) have shown that this can be achieved by the addition of inorganic powdered materials to the adhesives. This may be illustrated by the adjustment of the thermal expansion coefficient of polystyrene to that of brass.

Pure polystyrene has a thermal expansion of $70 \times 10^{-6}/^{\circ}C$; whereas that of brass is $17 \times 10^{-6}/^{\circ}C$. When brass inserts are placed in pure polystyrene, very small changes in temperature cause the plastic to crack and become detached from the metal. If 11 percent aluminum oxide, having an expansion coefficient of $8.7 \times 10^{-6}/^{\circ}C$, is added to the polystyrene, cracking does not occur over a wide temperature range and the bond remains intact.

This same principle is frequently employed in the use of carbon black in rigid adhesives. Carbon has a very low expansion coefficient (5.4×10^{-6}). When 10 percent is added to phenol-formaldehyde resin, the coefficient of expansion is 20 percent less than that of the pure resin. Equations for calculating the composition of adhesive mixtures required to obtain a desired thermal expansion coefficient are given in references 44 and 45.

B. TEST METHODS FOR BOND STRENGTHS

Comparatively little work has been done on the development and standardization of testing methods for determining the fundamental physical properties of adhesively bonded joints. The American Society for Testing Materials has

recently organized Committee D-14 on Adhesives to formulate methods of tests pertaining to adhesives. The cooperative efforts of the members of this committee should contribute to a better understanding of the problems involved in testing adhesively bonded joints and should provide a basis for obtaining uniform test data for bonds formed between the many available varieties of adhesives and adherends.

Some of the testing methods which have been used or proposed for the determination of bond strengths will be briefly reviewed to complete this survey on adhesives and adhesion.

1. Tensile Strength

A method for the determination of the tensile strength of glued joints based on experimental work at the Bell Telephone Laboratories (reference 46) has been prepared for consideration by the Subcommittee on Strength Properties of A.S.T.M. Committee D-14 on Adhesives. This method provides for the type of specimen shown in figure 6 and specifies that self-alining grips shall be employed in order to assure loading exactly normal to the glued surfaces.

2. Shear Tests

In the testing of glued joints in shear it is very difficult to attain pure shear conditions. A variety of methods have been proposed for the determination of this property. The block shear joint test and the plywood joint test are currently used in Army-Navy Aeronautical specifications. These and other proposed shear tests for glued joints are included in the following summary of such tests.

a. Block shear (reference 47).- The block shear test is the method most widely used for evaluating the bonding strength of adhesives to wood. The specimen and shearing tool are shown in figure 7. The specimen is broken by the application of a compressive load.

b. Plywood shear (reference 47).- The plywood shear test specimen consists of a three-ply wood laminate in which the grain of the center ply is at right angles to the two face plies. The specimen is prepared by milling a groove two-thirds through the core on each of the face plies. The specimen is broken under a tensile load in special grips as shown in figure 8.

c. Single lap joint shear (reference 48).- The single lap joint specimen has been widely used for the measurement of adhesion to metals. The specimen is broken under a tensile load. Standard grips for tensile specimens are used. The specimen is shown in figure 9.

d. Double lap joint shear (reference 48).- The double lap joint specimen was developed for the purpose of overcoming the unequal distribution of stresses encountered in the single lap joint specimen, and is broken in the same manner. The specimen is shown in figure 9.

e. Scarf joint shear (reference 48).- The scarf joint specimen is difficult to prepare, but the shearing stresses developed in the glue line are more uniform than for any other type of shear test. If the two adherends are of equal modulus, the single scarf joint is adequate. For materials of unequal modulus, however, it is necessary that the angle taper of each be proportional to its modulus, thus necessitating a double scarf joint. These joints are illustrated in figure 9. The specimens are broken under a tensile load.

f. Cylindrical single shear (reference 14).- The cylindrical single shear test was used by McBain to reduce the amount of wood failure in the glue line. The grips and specimen are shown in figure 10. By means of a special adjustment it is possible to vary the angle θ shown in the figure. It was found that the most reproducible values were obtained when $\theta = 60^{\circ}$. The specimen is broken under a compressive load.

g. Johnson double shear.- The Johnson shear test is performed with the specimens and shearing tool shown in figure 11. It consists in rigidly clamping the outer sections of the specimen in the shearing tool and applying a compressive load upon the shear member which applies an evenly distributed load to the central portion of the specimen. It is necessary that the two glue lines be accurately placed in the shearing planes of the instrument; thus very careful machining of the specimen is required for reproducible values.

The cylindrical double shear test used by McBain (reference 14) is similar to the Johnson shear test. The essential difference is that the cylindrical specimen is broken by a tensile load. The grips and specimen for this test are shown in figure 12.

h. Discussion of shear tests.- Each of these tests has

disadvantages; some give poor reproducibility and others require a high degree of accuracy in machining. The reasons for poor reproducibility may be illustrated by considering the single lap joint specimen. It has been shown (reference 49) that stresses applied to such a specimen are not equally distributed over the entire glue line, but are concentrated near the ends of the overlap. This is illustrated in figure 13.

This unequal stressing is due to the shape of the test piece rather than the means for applying the load. A given load will be concentrated excessively at the ends of each member on the overlap and very slightly on the exact center of the glued area. The result will be a partial shear failure and a partial peeling action. The shear value obtained in this way will not be directly proportional to the area of the overlap, but will reach a maximum value after which increasing the area will not change the shear value.

De Bruyne has found, however, that by tapering the ends of the overlapped sections, the shear value will become proportional to the area. A comparison of shear breaking loads obtained by these two methods is presented in figure 14.

It is shown in figure 9 that a similar modification of the double lap joint eliminates even more of the distortional error.

Shear methods based on torsion have not been investigated to any extent. These methods are also hampered by the problem of nonuniform stress distribution over the entire glued area. A rigorous consideration of the stress problems in shear testing has been presented by Goland and Reissner (reference 50), and also by Hearmon (reference 51).

National Bureau of Standards,
Washington, D. C., February 23, 1945.

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o. Vinyl Resins:

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2. Patents

a. Phenolic Resins.

1. Cresol - Formaldehyde
United States: 2,232,718; 2,271,744.

2. Phenol - Formaldehyde
United States: 2,186,369: 2,218,373; 2,229,357; 2,267,390; 2,287,536; 2,304,263; 2,309,610; 2,314,076; 2,317,364; 2,319,142.

British: 532,716; 540,404; 544,845.

French: 842,208; 845,339.

German: 699,605; 719,694; 725,499; 725,650.

3. Resorcinol - Formaldehyde
United States: 2,314,996.

b. Organic Nitrogen Resins:

1. Alkylolcyanamide
United States: 2,300,570.

2. Alkylolguanidine
United States: 2,298,473.

3. Bisthioammeline polyalkylene ether
United States: 2,325,105.

British: 546,373.

4. Ethylene Imine
German: 729,248.

5. Melamine - Aldehyde
United States: 2,318,121; 2,331,446.

British: 549,496.

German: 721,240.

6. Superpolyamides
United States: 2,224,037; 2,281,415; 2,296,555.

German: 721,187.

7. Urea - Formaldehyde
United States: 2,180,547; 2,190,239; 2,192,585; 2,193,630; 2,203,501; 2,223,536; 2,247,764; 2,275,821; 2,283,740; 2,287,756; 2,290,946; 2,303,982; 2,304,600; 2,306,057; 2,312,210; 2,314,308; 2,315,776; 2,320,301.

British: 512,659; 516,915; 521,653; 531,356; 536,493; 545,409.

French: 851,404.

German: 681,324; 681,372; 698,656; 722,218; 736,618.

c. Alkyd Resins:

United States: 2,194,013; 2,202,765; 2,215,219; 2,224,035; 2,230,230; 2,252,393; 2,280,256; 2,288,315.

British: 544,057; 547,328.

d. Furane Resins:

1. Furfuryl Alcohol - Formaldehyde
United States: 2,306,924.

2. Furfuryl Silicate
United States: 2,300,812

e. Polymerization Resins:

1. Acrylic Polymers

a. Acrylic Acid Esters
United States: 2,214,158; 2,221,367; 2,280,981.

German: 575,327; 705,520.

b. Methacrylic Acid Esters
United States: 2,199,597; 2,231,471; 2,268,611; 2,280,981, 2,281,589.

German: 697,083; 705,329.

2. Ethylene Polymers
United States: 2,219,684; 2,263,249.

British: 544,359.

3. Styrene Polymers
United States: 2,276,478.

British: 522,553.

German: 698,656.

4. Vinyl Acetal Polymers
United States: 2,197,420; 2,200,969; 2,205,020; 2,213,751; 2,222,490; 2,227,983; 2,232,806; 2,233,941; 2,240,027; 2,274,672; 2,279,145; 2,279,901; 2,317,891; 2,327,652.

British: 525,556.

Canadian: 397,250; 397,476.

German: 690,332.

5. Vinyl Alcohol Polymers
United States: 2,234,829; 2,242,019.

German: 704,462.

6. Vinyl Ester Polymers
United States: 2,168,220; 2,209,435; 2,219,433; 2,259,490; 2,319,826; 2,322,048.

Australian; 108,199.

British: 510,826; 511,996; 512,478; 542,658; 542,964.

German: 687,486; 705,520; 731,932.

7. Vinyl Ether Polymers
United States: 2,300,587.

French: 849,442.

German: 705,394.

8. Vinyl Halide Polymers
United States: 2,261,313; 2,300,587.

French: 845,569.

German: 698,655; 713,793.

9. Vinyl Lactam Polymers
United States: 2,265,450.

f. Cellulose Derivatives:

1. Cellulose Esters
United States: 2,176,837; 2,188,395; 2,214,286; 2,223,575; 2,234,236; 2,258,991; 2,263,661; 2,281,483; 2,296,891; 2,324,097; 2,324,680; 2,325,700.

British: 514,172.

Canadian: 395,145; 400,105.

German: 681,322; 707,659; 730,306.

Russian: 52,331.

2. Cellulose Ethers
United States: 2,171,140; 2,210,704; 2,224,523; 2,273,677; 2,285,178; 2,319,834; 2,329,741; 2,330,313.

British: 529,993.

Canadian: 398,320; 400,643.

French: 841,836; 841,885.

German: 716,221; 735,864.

g. Carbohydrates (Except Cellulose Derivatives)

1. Dextrins
United States: 2,172,357; 2,174,541; 2,181,782; 2,188,099; 2,190,372; 2,204,384; 2,210,119; 2,241,700; 2,287,599; 2,303,791; 2,304,730.

2. Gums
United States: 2,220,988; 2,319,102.

3. Starches
United States: 2,194,216; 2,202,678; 2,212,557; 2,215,847; 2,215,849; 2,221,484; 2,222,872; 2,222,873; 2,222,874; 2,222,875; 2,238,767; 2,258,741; 2,275,314; 2,282,364; 2,283,044; 2,321,072.

British: 511,026; 527,704; 543,432; 543,433.

Canadian: 403,788.

French: 852,825; 852,826; 852,827.

German: 719,568.

h. Proteins:

1. Animal Glue
United States: 2,170,273; 2,176,038; 2,182,425; 2,214,231; 2,226,553; 2,235,202; 2,246,405; 2,265,144; 2,282,177; 2,308,185; 2,320,087; 2,323,831.

British: 505,868.

Canadian: 394,976; 413,995.

German: 700,759.

2. Blood Albumin
United States: 2,180,542; 2,307,198.

3. Casein
United States: 2,180,542; 2,180,543; 2,182,357; 2,197,168; 2,300,907; 2,302,378; 2,330,428; 2,332,519; 2,334,270.

Australian: 110,458.

British: 531,084; 540,326; 543,302; 543,325.

4. Chitin
United States: 2,201,762.

German: 716,428.

5. Seed Meal
United States: 2,174,438; 2,233,213; 2,243,871; 2,297,340; 2,312,056; 2,332,801.

6. Soybean
United States: 2,178,566; 2,260,640; 2,271,620; 2,274,983; 2,284,700.

Japanese: 134,071, 134,130.

7. Zein
United States: 2,229,028; 2,250,040.

Canadian: 406,768.

German: 716,340.

i. Rubbers:

1. Butadiene Polymers
United States: 2,265,722; 2,273,880.

Russian: 53,402.

2. Chloroprene
United States: 2,212,611; 2,286,505; 2,300,352; 2,328,351.

3. Cyclized Rubber
United States: 2,300,368.

4. Halogenated Rubber
United States: 2,234,621; 2,259,190.

British: 524,070.

5. Isobutylene Polymers
United States: 2,213,331; 2,226,589; 2,319,959.

French: 849,983.

6. Natural Rubber
United States: 2,203,677; 2,209,451; 2,226,938; 2,232,791; 2,240,862; 2,254,321; 2,256,194; 2,257,083; 2,259,350; 2,263,305; 2,269,660; 2,277,992; 2,278,355; 2,278,609; 2,279,110; 2,281,087; 2,284,947; 2,297,837; 2,295,866; 2,297,871; 2,300,592; 2,304,678; 2,310,972; 2,311,301; 2,318,126; 2,335,104.

British: 514,037; 517,816; 521,108; 521,401; 522,057; 523,152; 531,203; 533,630; 542,331, 547,841; 551,398.

Canadian: 391,000; 393,229; 393,230; 400,442; 407,463; 409,651; 415,026; 415,030.

Dutch: 49,687.

French: 844,367.

Russian: 54,276; 57,435.

Swiss: 213,019.

7. Phenol - Formaldehyde Modified Rubber
United States: 2,314,997; 2,343,551.

British: 530,697; 544,944.

Canadian: 415,029.

8. Neoprene
United States: 2,196,602; 2,226,541; 2,313,039.

9. Rubber Hydrochloride
United States: 2,174,673; 2,218,617; 2,320,716.

10. Sulfide Polymers
United States: 2,206,643; 2,252,366.

German: 707,438; 709,691.

j. Miscellaneous Natural Organic Products:

1. Asphalt
United States: 2,175,767; 2,280,653; 2,283,937; 2,286,244; 2,290,833; 2,322,886.

British: 533,927.

Canadian: 405,350.

2. Bitumins
United States: 2,280,699.

British: 519,119; 519,463; 538,105.

French: 847,749; 847,829; 850,940.

3. Fatty Acid Salts
United States: 2,284,570.

Canadian: 415,383.

German: 694,158.

Japanese: 128,323; 129,852.

4. Lecithin
British: 525,250.

5. Pitches
Unites States: 2,208,580; 2,218,335; 2,288,293.

British: 533,546.

German: 736,874.

Russian: 54,518; 55,150.

6. Rosin Esters
United States: 2,179,339; 2,223,086; 2,285,416; 2,307,801; 2,319,271.

German: 710,324.

Russian: 55,670.

7. Wood Waste Derivatives
United States: 2,294,666; 2,319,883.

8. Waxes
German: 702,740; 704,088; 704,754.

Russian: 52,976.

Swiss: 209,644.

k. Inorganic Substances:

1. Boron Compounds
United States: 2,219,583; 2,223,349; 2,231,718.

Canadian: 390,041.

2. Phosphorus Compounds
British: 521,694.

3. Silicon Compounds
United States: 2,215,048; 2,239,358; 2,261,784; 2,278,345; 2,285,053; 2,287,410.

Belgian: 440,870.

British: 526,462; 529,593; 542,655; 552,143.

German: 688,575; 698,389; 701,503.

3. Aircraft Manufacturers' Reports

a. Allied Aviation Corp., Cockeysville, Md.

1. Tests on Thermosetting Adhesives for Rubber Bag Cooking.

2. Test Program for Durez Resin 11814.

3. Shear Block Tests - Cascophen Resins.

4. Tests of Cast Durez Resin.

5. Tests for Special Assembly Using Cold Setting Durez 11814.

6. Shear Block Tests Using Cold Setting DuPont Adhesive J4600 X 5100.

7. Test on Melmac 401.

8. Shear Block Tests on 8 Miscellaneous Adhesives.

9. Shear Block Tests on Durez 11814 with Catalyst 7422.

10. Comparison of Various Percentages of No. 7422 Accelerator-Durez 12041.

11. Shear Block Tests Using Durez Resin 12041.

12. Reports and Graphs on Miscellaneous and Sundry Tests.

E R R A T U M

NACA TN No. 989

SURVEY OF ADHESIVES AND ADHESION
By R. C. Rinker and G. M. Kline
August 1945

Page 73: Insert the heading

"Boeing Airplane Co., Seattle, Washington"

between reports 11 and 12.

b. Beech Aircraft Corp., Wichita 1, Kansas:

1. Resin Glue Specification for Wood Gluing.

2. Cementing Similar and Dissimilar Materials - Sealers, Non-Skid Walkway and Floor Material.

c. Boeing Airplane Co., Wichita, Kansas:

1. A Comparison of Commercial Rubber Cements for Bonding Insulation to Wood or Metal. Rep. No. 841.

2. Tests on Cascamite Cold-Setting Resin Glue. Rep. No. 6341.

3. Gluing to a Glue-Coated Surface - Casein Glue. Rep. No. 6354.

4. Hot Press Gluing of Laminated Spars, Scarf Joints and Ribs. Plaskon 250-2. Rep. No. 6372.

5. Tests on One-Tenth Inch Laminated Fir Spar Stock. Rep. No. 6375.

6. Fatigue Tests on Wood and Glued Wood Assemblies. Rep. No. WD-10464.

7. Specification for Hot Press Gluing Spars. Rep. No. WD-11051.

8. Gluing of Paper Base Phenolic Plastic. Rep. No. WD-11101.

9. Specification for Gluing Paper Base Phenolic Plastic. Rep. No. WD-11102.

10. Cement for Bonding Insulation Material to Metal. Rep. No. WD-11104.

11. Amberlite PR-75B; Low Temperature Phenol-Formaldehyde Glue. Rep. No. WD-11105.

12. Technical Information on Adhesives. Rep. No. D-3790.

13. Boeing Process Specification for General Application of Cements and Glues. Rep. No. BAC 5010.

14. Boeing Process Specification for Joining and Insulating Air Ducts. Rep. No. BAC 5403.

15. Boeing Process Specification for the Handling and Cementing of Methyl Methacrylate Enclosures with Synthetic Rubber Mountings. Rep. No. BAC 5410.

16. Design Manual, Sec. 12.11. Rep. No. D-5000.

17. Miscellaneous Tests on Synthetic Resin Adhesives. Rep. No. T-24549.

d. Bell Aircraft Corp., 2050 Elmwood Ave., Buffalo 7, N.Y.:

1. Cycle-Welding 24S-T Alclad to Birch. Rep. No. BTR 43-86.

e. Chance Vought Aircraft, Stratford, Conn. (Division of United Aircraft Corp.)

1. Rubber Cements - Commercial and Synthetic Bonding Neoprene Sheeting to Aluminum Alloys. Rep. No. 38.

2. Cements - Rubber or Neoprene to Metal (Gasoline and Oil Resistant). Specification No. 99.

3. Wood Gluing; Cold Setting Urea. Specification No. 110.

4. General Purpose Cement. Specification No. 111.

5. Application of Fabric to Metal or Plywood Surfaces. Specification No. 113.

6. Attachment of Walkways. Specification No. 114.

f. Consolidated Vultee Aircraft Corp., San Diego 12, Calif.:

1. Development and Use of Metlbond. Rep. No. ZM-223.

g. Curtiss-Wright Corp., Louisville, Ky.

1. Material Substitution at Curtiss-Wright. Rep. No. GI-104-D2.

2. Preparation and Application of "Weldwood" Plastic Glue. Rep. No. L3-12-1

3. Cementing Procedure - Antenna Reflectors. Rep. No. L-3-12-2.

4. Accelerated Gluing of Scarf Inlays. Rep. No. L-3-12-3.

5. Cementing Flap Seals (CE 10003) to Flap Closures. Rep. No. L-3-12-4.

6. Cementing of Gap Closures on C-76 Airplanes. Rep. No. L-3-12-5.

7. Shop Procedure for the Use of Infra Red Heat Lamps in Curing Glue Bonds. Rep. No. L-3-12-6.

8. Haskelite Moulded Part Using Dry-Film Glue. Rep. No. L-43-6.

9. Accelerated Curing of Plaskon 250-2 Glue. Rep. No. L-43-14.

10. Glue Bonds Floor and Side of Ship No. 7. Rep. No. L-43-25.

11. Comparison of Elastic Cements. Rep. No. L-43-35.

12. U.S. Rubber Co., No. 355 Cement. Rep. No. L-43-36.

13. Cold Setting National Liquid Resin Glue. Rep. No. L-43-61.

14. Bakelite Urea Resin with Catalyst. Rep. No. L-43-64.

15. Investigation of Lauxite 77-X. Rep. No. L-43-69.

16. Plaskon Resin Glue 201 and 250-2. Rep. No. L-43-70.

17. Cascamite Urea Resin Glues. Rep. No. L-43-71.

18. M and MT Miracle Adhesives. Rep. No. L-43-77.

19. Fairpreen Caulking Compound and Cement. Rep. No. L-43-81.

20. Uformite Resin Adhesives CS-551 and 552. Rep. No. L-43-82.

21. Le Pages Panite Plastic Resin Glue. Rep. No. L-43-84.

22. Perkins Resin Glue. Rep. No. L-43-87.

23. Glue Film Thickness on pH. Rep. No. L-43-88.

24. Uformite Resin Glue No. 500. Rep. No. L-43-90.

25. Uformite Resin Glue No. 430. Rep. No. L-43-92.

26. Hot Press Cascamite 66 with Catlyst H-19. Rep. No. L-43-96.

27. Hot Press Uformite 500 with Catlyst Y. Rep. No. L-43-97.

28. Effect of Drying Time on pH of Glue Film. Rep. No. L-43-100.

29. Riveted Panel Reinforced with Thermoset Adhesive. Rep. No. NE-93-S1.

30. Plastics in the P-40 Box. Rep. No. PC-227-D1.

31. C-46 Floor Door. Rep. No. PE-127-D1.

32. C-46 Pedestal Control Knobs. Rep. No. PE-217-D1.

33. An Approximate Elastic Solution for the Jointing of Two Similar Plates by Adhesion. Rep. No. RP3-S1.

34. Tests of C-76 Yellow Poplar Plywood. Rep. No. SH-106-S1.

35. Tests of C-76 Yellow Poplar Plywood. Rep. No. SH-106-S2.

36. The Small Bending Theory for Flat Laminated Plates. Rep. No. 5S-13.

37. Static Test of a Plywood Floor Panel. Rep. No. 5S-14.

38. Results Obtained in the Gluing of Compreg. Rep. No. 5S-15.

39. Acidity of Cold-Setting Urea Resin Adhesives. Rep. No. 5S-16.

40. Solubility of Resin Adhesives. Rep. No. 5S-17.

41. "Free" Water in Urea Resin Adhesives. Rep. No. 5S-18.

42. Gluing Resin Sealer Coated Wood. Rep. No. 5S-19.

43. Progress Report on the Bonding of Aluminum Alloy. Rep. No. 5S-20.

44. Alkaline Contamination of Resin Glues. Rep. No. 5S-21.

45. Control of Wood Aircraft Manufacture and Inspection. Rep. No. 5S-23.

46. Properties of Artificial Resin Adhesives. Rep. No. 5S-26.

47. Effect of Catalyst Proportion upon the Acidity. Rep. No. 5S-28.

48. The Veneer Orientation for Plywood Plates. Rep. No. 5S-57.

49. A Preliminary Investigation of Lignin and Its Derivative Products. Rep. No. WA-132-S1.

50. The Effect of the Bag Molding Process upon the Strength of Low Density Plywood. Rep. No. WB-171-M1.

51. Cold Setting Phenolic Adhesives. Rep. No. WE-11-M9.

52. Fatigue of Resin Bonded Wood Joints. Rep. WE-11-M10.

53. Test of Resinox No. 840 (A Melamine Resin Film Adhesive) Rep. No. WE-11-S-1.

54. Test of Resinox No. 841 (A Melamine Resin Film Adhesive) Rep. No. WE-11-S-2.

55. Test of Penacolite, A Cold Setting Resorcinol Adhesive. Rep. No. WE-11-S4.

56. Properties of Urea Resin Adhesives. Rep. No. WE-11-S-5.

57. Properties of Artificial Resin Adhesives. Rep. No. WE-11-S-6.

58. Test of Du Pont Adhesive No. 4624. Rep. No. WE-11-S-7.

59. Test of Bakelite XCu - 16257 (Hardener XK 16229). A Craze Resistant Urea Resin Adhesive. Rep. No. WE-11-S-8.

60. Setting Rates of Urea Resin Glues. Rep. No. WE-15-S1.

61. Low Temperature Phenol Formaldehyde Adhesives. Rep. No. WE-16-S1.

62. Evaluation of Moderate and Low Temperature Adhesives, Du Pont 4631-J-85-0025 and Bostik T-46-M (A-300). Rep. No. WE-18-M6.

63. Test of Resinox No. 410, Wood to Metal Adhesive. Rep. No. WE-18-S1.

64. Test of Resimul No. 697, Wood to Metal Adhesive. Rep. No. WE-18-S2.

65. Bonding of Metal to Wood or Plastics with Bostik Adhesives. Rep. No. WE-18-S3.

66. C-76 Stringer Splice Fitting Bonded with Wood to Metal Adhesive. Rep. No. WE-18-S4.

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4. Melmac Adhesive 400.
5. The Beetle Cements A and AF.
6. Laminac Resin P-4122.

b. Bakelite Corporation, 30 East 42d St., New York 17, N.Y.:

1. Powdered Phenol Resin Glue BC-16168.
2. Powdered Phenol Resin Glue BC-16529.
3. Bakelite Cold Press Wood Bonding.
4. Warm-Setting Phenolic Resin Glue BC-17540.
5. Cold-Setting Phenolic Resin Glue XC-17613.

c. B.B. Chemical Co., 784 Memorial Drive, Cambridge 39, Mass.:

1. Physical Tests on Aluminum to Aluminum Bonds.

d. Carbide and Carbon Chemicals Corporation, 30 East 42d St., New York 17, N.Y.:

1. Vinylite Polyvinyl Acetate Resins.

2. "Vinylseal" Adhesives T 24-9 and MA 28-18.

e. Casein Company of America, 350 Madison Avenue, New York 17, N.Y.:

1. Wood and Glue at War.

2. The Story of Cascamite, America's New Plastic Glue.

3. Glued Prefabricated Houses.

4. Casco Gluing Guide.

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15. Bulletin No. 6. Glue Recommendations for "Fluid Pressure" or Bag-Gluing Techniques.

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19. Bulletin No. 10. Spraying Synthetic Resin and Casein Glues.

20. Bulletin No. 11. A Cold-Setting Cement for Bonding Metal to Wood and Other Dissimilar Materials (Casco Flexible Cement NT-442)

21. Bulletin No. 12. A Durable Flexible Cement of Wide Application (Casco Flexible Cement NT-475)

22. Bulletin No. 13. Edge Gluing with Urea Resin Glue in the Rotary Clamp.

23. Bulletin No. 14. Cleaning Glue Equipment.

24. Bulletin No. 15. Thinning Intermediate - Temperature - Setting Phenol Resin Glues.

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26. Bulletin No. 17. Cascamite 66-Cold Press with Flour Extender.

27. Bulletin No. 18. Casco-Resin 5 (Liquid)-Cold Press with Flour Extender.

28. Bulletin No. 19. The "Master Mix" Method of Using Urea Resin Glues.

29. Bulletin No. 20. A New, Room-Temperature-Setting Resin Glue Producing Durable, Boilproof Wood Joints.

30. Directions for Using Casco Resin for Hot-Press Gluing.

31. Casco Resin (Liquid) (Use with Catalyst H-19)

32. Cascamite TS - Cascamite TS-75.

33. Cascamite BG-2 (Use with Catalyst H-21)

34. Cascamite LA-27.

35. Cascamite 66 (Use with Catalyst H-19).

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37. Cascophen LT-67 (Liquid Phenol-Formaldehyde Resin Glue).

38. Cascophen W-110.

39. Cascophen P-48.

40. Cascophen BG-17 (Use with Catalyst H-23).

41. Compregnite.

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g. Chrysler Corporation, Detroit 31, Mich.:

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2. Moisture and Humidity Resistance of the Cycle-Bond Process.

3. Cycle-Bond (Preliminary Report).

4. CB-2 Plywood Shear Immersion Tests.

5. Cycle-Weld CB-4 Process VS C-3 Cement.

6. High and Low Temperature Shear Tests on Cycle-Welded Alclad and Dow Metal Sheet.

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8. Chrysler Direction Sheets and Process Standards.

9. Cycle Weld Chart.

10. Cycle Weld, Bonding of Wood to Metal.

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2. Lucite Manual - Methyl Methacrylate Resin.

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4. 4644 Thermoplastic Adhesive.

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6. 4646 Modified Thermoplastic Adhesive (Restricted).

7. 4653 Low Temperature Bonding Adhesive (Restricted).

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2. Durez 194 Hot Press Resin Adhesive

3. Durez 194 Resin Adhesive for Bag Molding.

4. Durez Low Temperature Resin Adhesive (12041 with Catalyst 7422)

5. Durez Low Temperature Alkaline Resin Adhesive (Tentative Instructions for the Use of 12533 with 12534B)

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3. Special Data No. 1064, Durite S-1643, Fabric Impregnant for Molded Shapes, May 1, 1944.

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7. Special Data No. 1073, Durite S-2988, Boil-Proof Thermosetting Resin for Treating and Laminating Fabric, Paper, etc., June 8, 1944.

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9. Data No. 4 Adhesives of S-2979 Series, June 23, 1944.

k. Monsanto Chemical Co., Plastics Division, Springfield 2, Mass.:

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l. Miracle Adhesives Corp., 261 Fabyan Place, Newark 8, N.J.:

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2. Directions for Installing Safety Treads (Specification M-202)

3. Recommended Spraying Equipment for Miracle Adhesive, Type P. Directions for Spraying Miracle Adhesive, Type P. (Specification M-203)

4. Directions for Applying Miracle Adhesive, Type P, with a Brush (Specification 203-A)

5. Directions for Installing Asbestos Fabric, Asbestos Listing, etc. (Specification M-204)

6. Directions for Installing Canvas (Specification M-205)

7. Directions for Installing Cork and Heavy Density Fibreglass Insulation (Specification M-206)

8. Directions for Installing Light Density Fibreglass and Batt Type Insulation (Specification M-207)

9. Directions for Installing Linotile (Specification M-208)

10. Directions for Installing Linoleum (Specification M-209)

11. Directions for Installing Wall Fixtures (Specification M-210)

12. A Leveling Plastic for Use under R-Mir-Dek, Tile, Linoleum, Linotile, etc. (Specification M-212)

13. Directions for Installing Cork, Insulation Board, Asbestos Board and Hard Board (Specification M-220)

14. Directions for Installing Glass Blocks (Specification M-221)

15. Directions for Installing Acoustical Board, Insulation Board, etc., to Ceilings (Specification M-222)

16. Directions for Installing Rubber with Miracle Adhesive, Type R (Specification M-250)

17. Directions for Installing Light Fabrics, Asbestos, Paper, etc., with Miracle Adhesive, Type R (Specification M-251)

m. Pennsylvania Coal Products Company, Petrolia, Pa.:

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2. Instruction Sheet, Penacolite G-1131.

n. Perkins Glue Company, Lansdale, Pa.:

1. Perkins Resin Glues, Handbook, Fourth Edition.

2. Directions for Using Perkins Aircraft Casein Veneer Glue.

3. Perkins Urea-Formaldehyde and Melamine-Urea Resin Glues. General Instructions.

   a. Cold Pressing Technique
   b. Hot Plate Pressing Technique

4. Perkins L-100 with Boil-Proofing Catalyst (for Vacuum Bag-Autoclave Application)

5. Perkins D-111 with Boil-Proofing Catalyst (for Vacuum Bag-Autoclave Application)

6. Perkins Melamine-Urea Resin M-411 (Instructions for Vacuum Bag-Autoclave Application)

7. Perkins Liquid Urea-Formaldehyde Resin -L-100.
   a. Cold Pressing
   b. Hot Plate Pressing

8. Perkins L-100 with Addition of Boil-Proofing Catalyst. (Hot Plate Pressing)

9. Perkins Powdered Urea-Formaldehyde Resin D-111.
   a. Cold Pressing
   b. Hot Plate Pressing

10. Perkins Powdered Melamine-Urea Resin M-411.
    a. Cold Pressing, but Kiln Cure
    b. Hot Plate Pressing

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o. Plaskon Division, Libby - Owens - Ford Glass Co., 2112-24 Sylvan Avenue, Toledo 6, Ohio:

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2. Plaskon Cold Setting Resin Glue, Type 201.

3. Plaskon Cold Setting Resin Glue, Instruction Manual for Ready-Mixed, Type 250-2.

4. Plaskon Hot Setting Resin Glue, Type 107.

5. Plaskon Resin Glue, Instruction Manual for Type 700-2 Hot Setting Adhesive.

p. Plastics Industries Technical Institute, 186 South Alvarado St., Los Angeles 4, Calif.:

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q. Resinous Products and Chemical Co., Washington Square, Philadelphia 5, Pa.:

1. Resin Adhesives for Plywood, Tego, Amberlite, and Uformite (7th ed.), Oct. 1942.

2. Bulletin No. 2. Uformite CB-551 Cold Bonding Studies. April 1943.

3. Bulletin No. 3. Amberlite PR-14 Hot Pressing Studies, May 1943.

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8. The Uformites, Cold Bonding Resin Adhesives.

9. Uformite CB-551 and Uformite CB-552, Cold Setting Resin Adhesives.

10. Catalyst Q-107 with Uformite 430 and Uformite 500.

11. Redux, for Bonding Metal to Metal.

12. Redux, for Bonding Metal to Metal and Metal to Wood.

13. Tego Tape, for Repairing Plywood Veneers.

14. Material 1142.

15. Edge Gluing of Lumber Cores with Uformite 500 and Catalyst Q-87.

r. Swift and Co., 4115 Packers Ave., Chicago 9, Ill.:

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4. Gabriel, A. E., and Cohodas, Leah E.: Analysis for Filler Content of Urea-Formaldehyde Glues. Rep. No. 1333, Sept. 1944.

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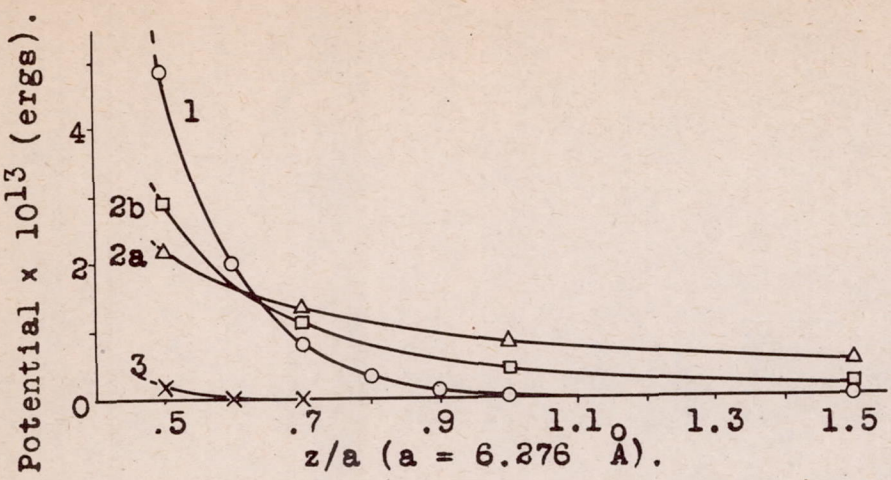


Figure 1.- The potential field outside a (100) plane of KCl on a charged particle (curve 1) and on argon (curve 2). (From Lennard-Jones and Dent).

Curve 1.- Electrostatic force between the crystal and charged particle.
Curves 2a and 2b. Van der Waals forces between the crystal and an argon atom.
Curve 3.- Force due to polarization of the crystal by the charged particle.
The forces are calculated for a particle at a distance "z" from the crystal surface in which the ions are separated by the distance "a".

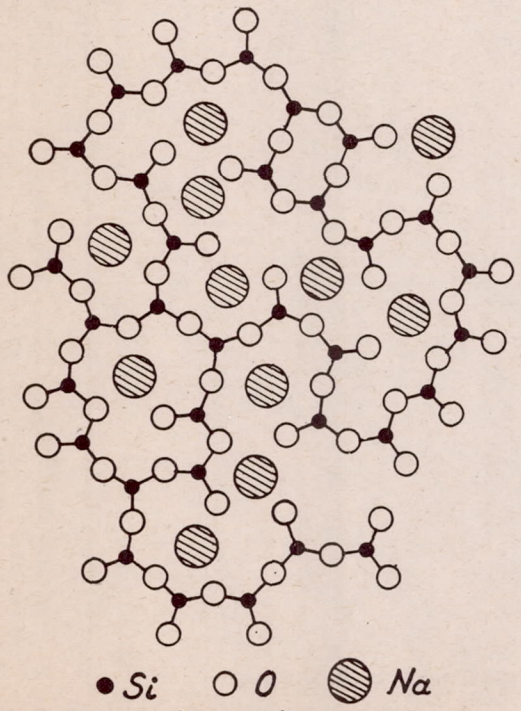


Figure 2.- Schematic representation in two dimensions of the structure of soda-silica glass. (From Warren).

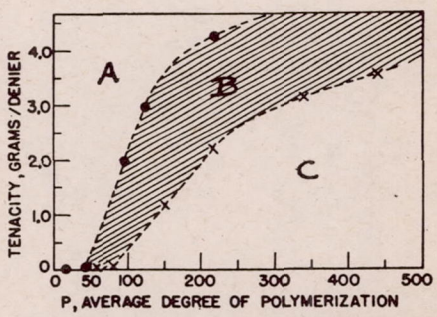


Figure 3.- Effect of average degree of polymerization on tenacity of high polymers. (From Mark).

A.- Fibrous area
B.- Plastic area
C.- Rubbery area

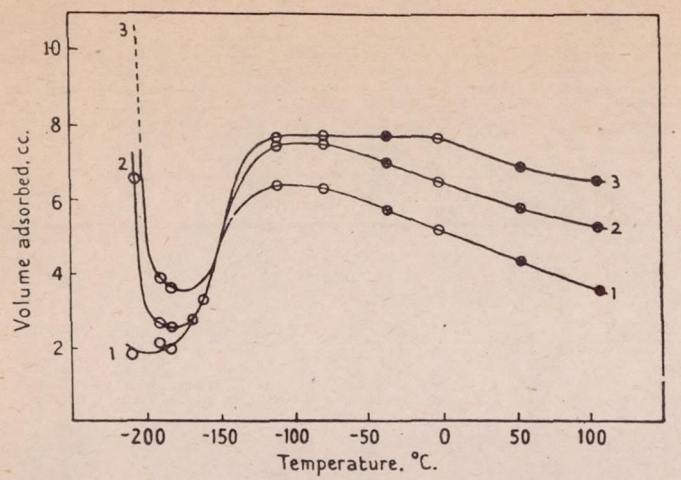


Figure 4.- Adsorption of hydrogen on nickel. (From Benton and White).

Curve 1.- 25 mm pressure
Curve 2.- 200 mm pressure
Curve 3.- 600 mm pressure

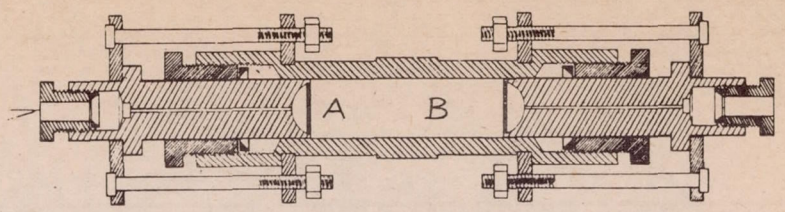


Figure 5.- The Bartell cell. (From Bartell and Osterhof).
A.- Section wet with highly wetting liquid.
B.- Section wet with poorly wetting liquid.

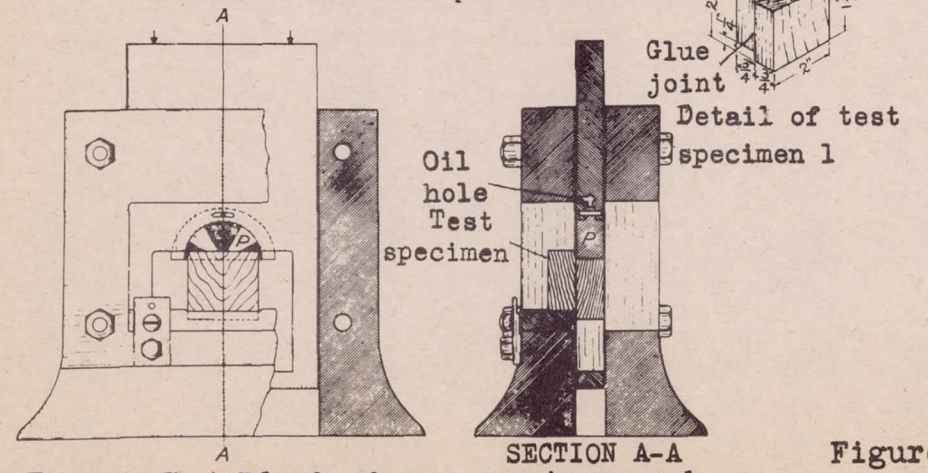


Figure 7.- Block shear specimen and tool. (From ANC-19).

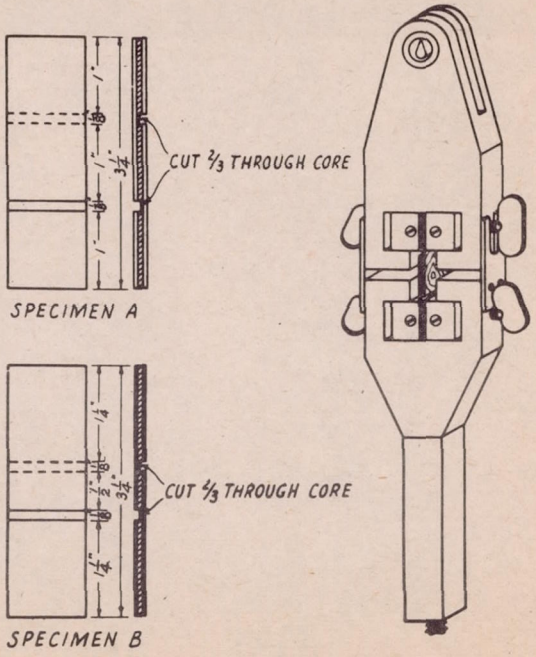


Figure 8.- Plywood shear specimens and grips. (From ANC-19).

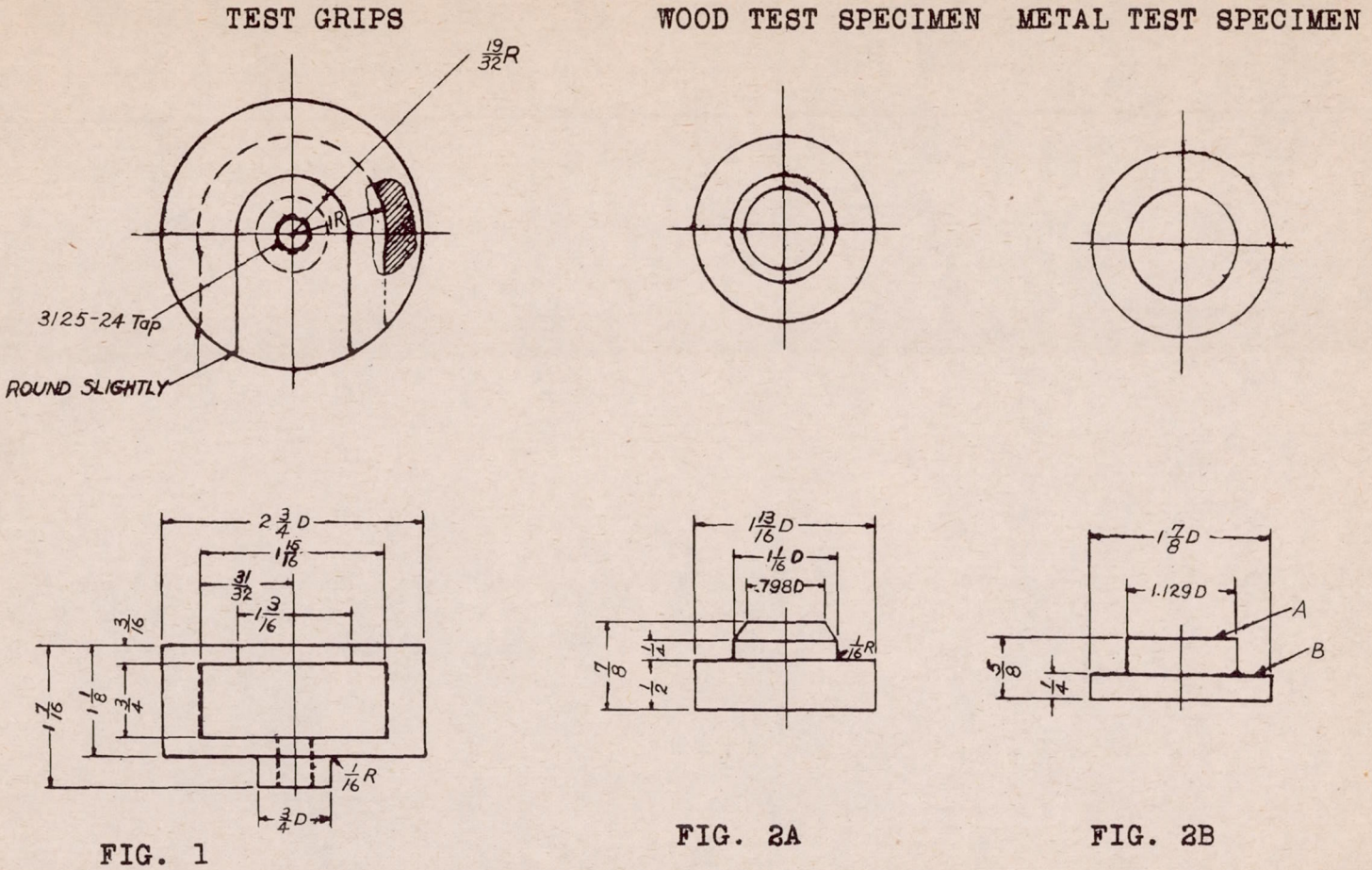
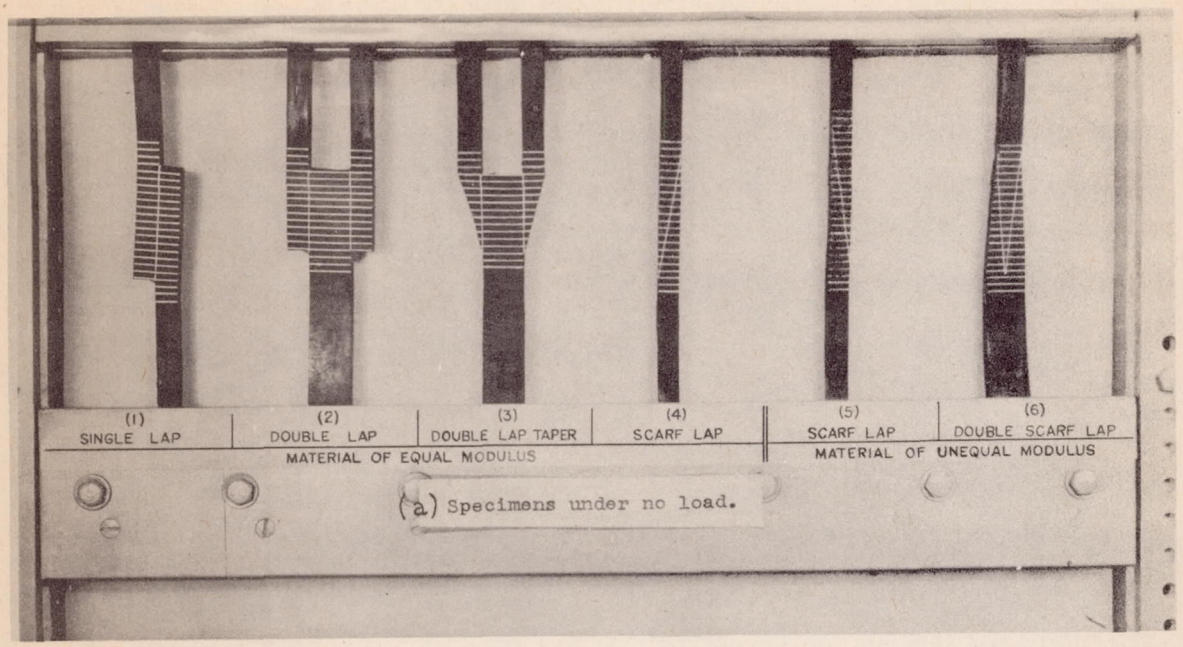


FIG. 1

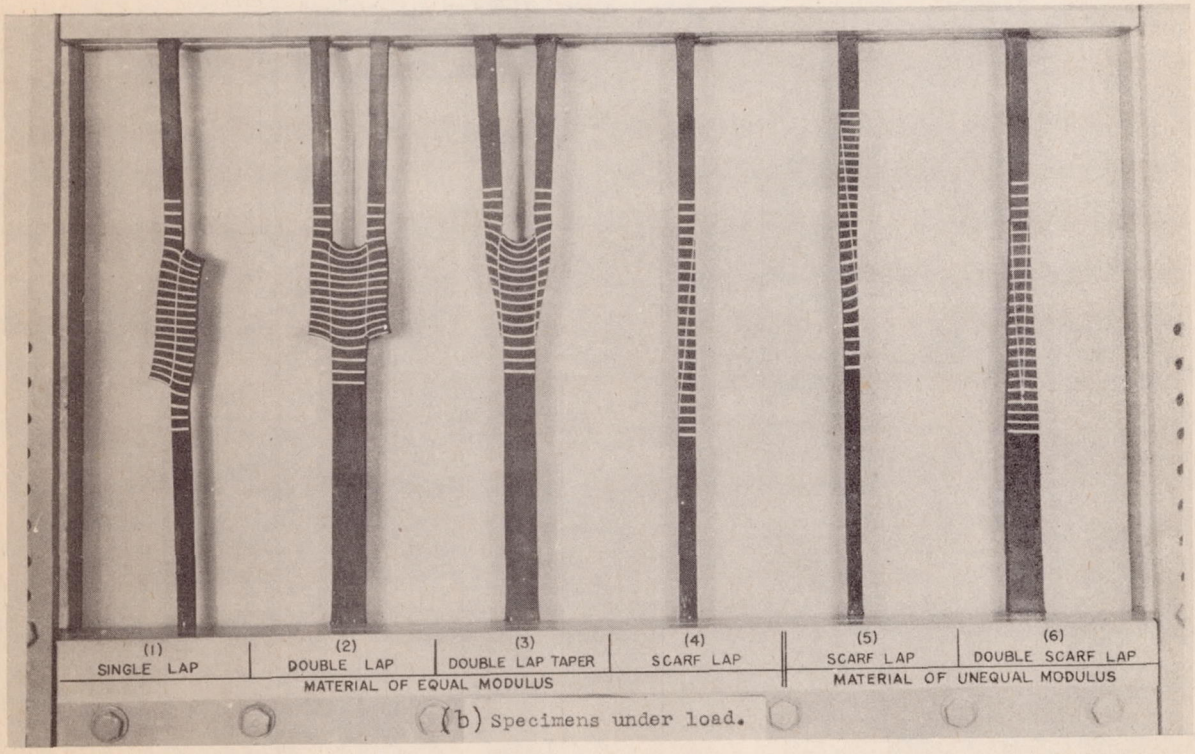
FIG. 2A

FIG. 2B

Figure 6. - Tensile test specimen (From Platow, reference 46).



(a) Specimens under no load.



(b) Specimens under load.

Figure 9.- Distribution of stresses in shear test specimens. (From Jackson).

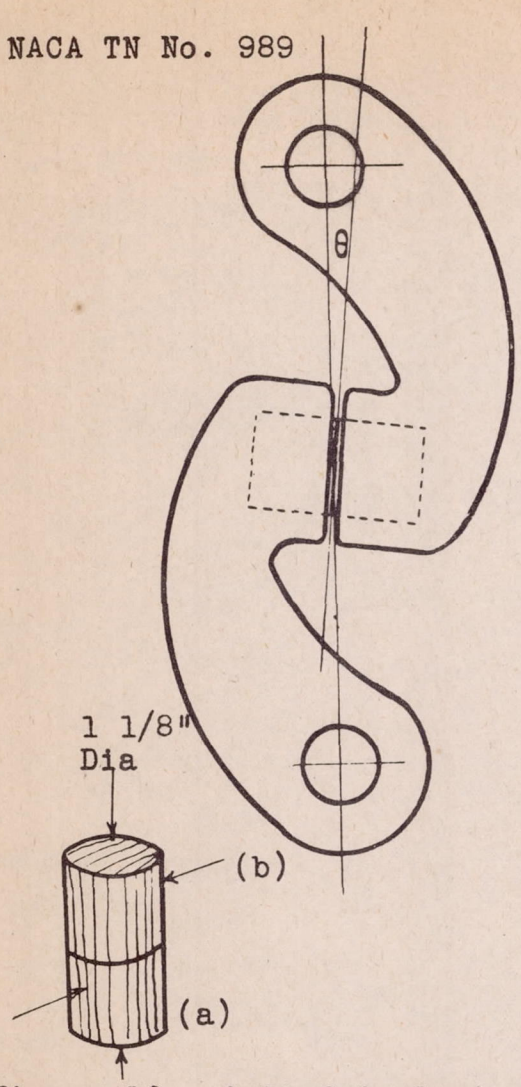


Figure 10.- Cylindrical single shear specimen and grips. (From McBain).

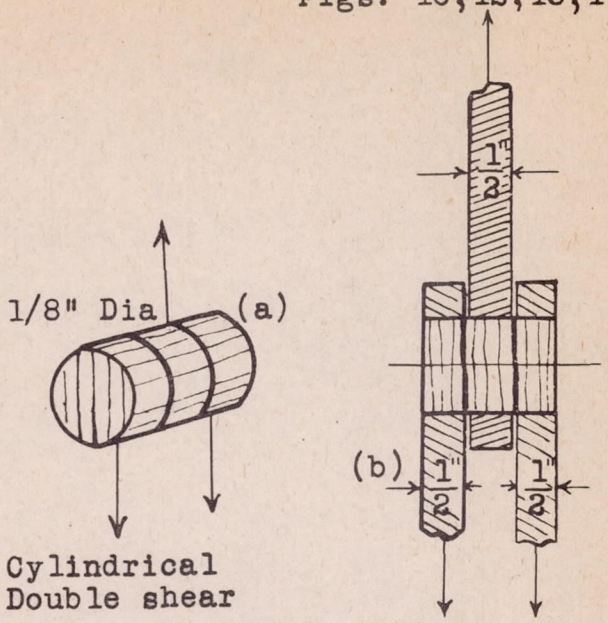


Figure 12.- Cylindrical double shear specimen and grips. (From McBain).

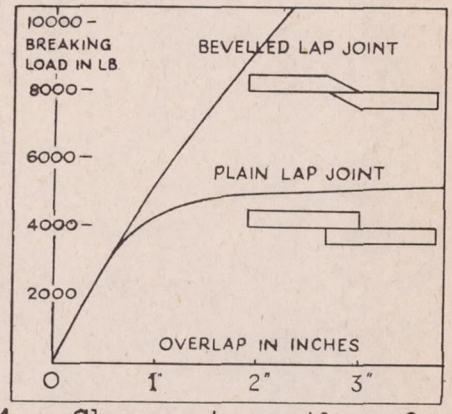


Figure 14.- Shear strengths of tapered and untapered lap joint specimens. (From de Bruyne)

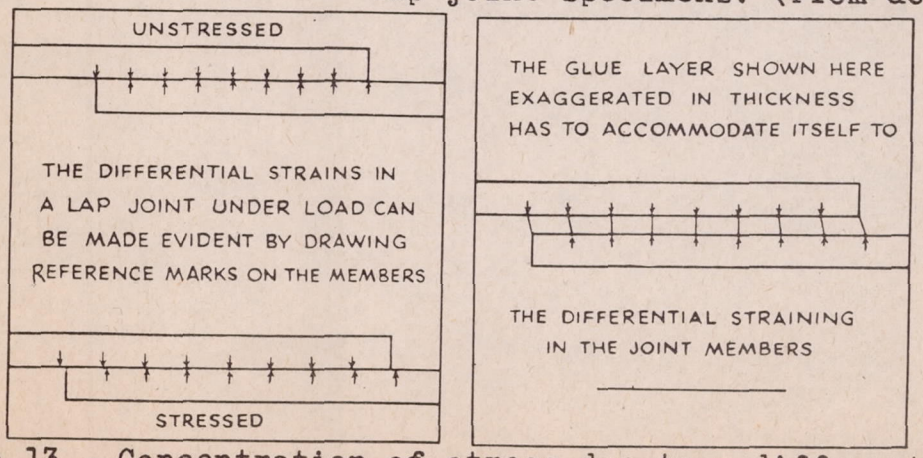
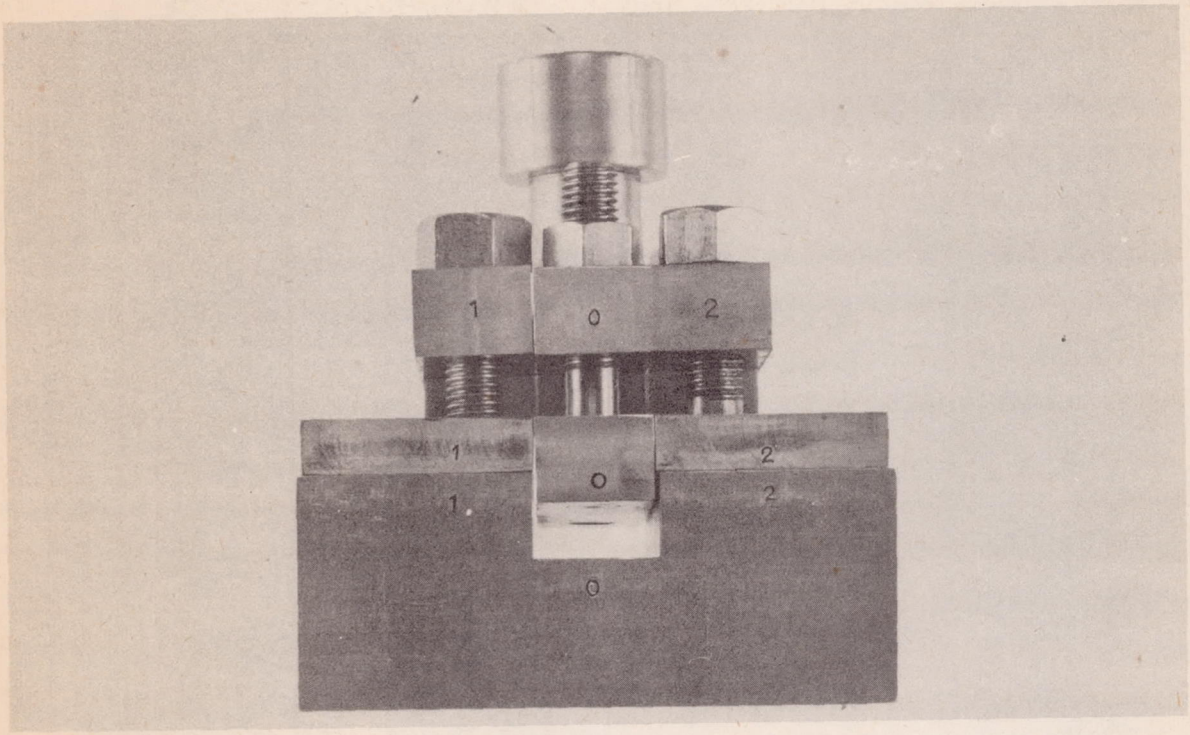


Figure 13.- Concentration of stress due to a differential strain in a lap joint specimen. (From de Bruyne).



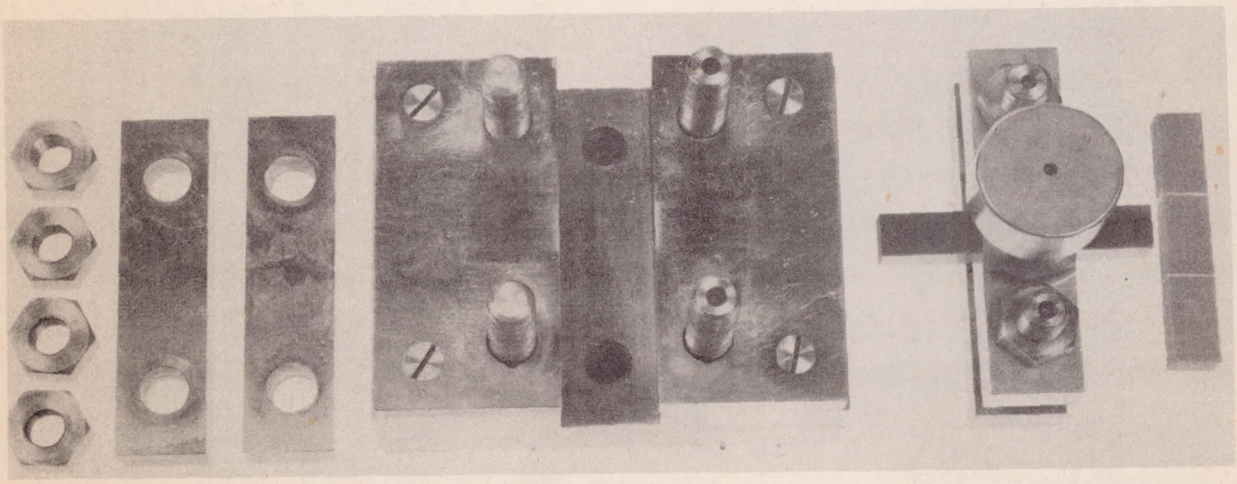


Figure 11.- Johnson double shear specimen and tool.