Joining of Polymers to Metal

The joining of high-strength thermoplastics to aluminum is one phase of an ongoing study of polymer-to-metal joints

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ABSTRACT. This paper reviews the factors controlling adhesion in polymer/aluminum and in polymer/steel joints, and the ongoing research being carried out at the University of Toronto on aluminum/oriented high-strength polypropylene bonding. Two approaches have been taken when examining the joining of aluminum to high-strength oriented polypropylene, *viz.*, electromagnetic induction and ultrasonic bonding (using specially formulated tie-layer materials). The lap-shear strength produced using electromagnetic and ultrasonic bonding exceeded the interfibrillar cohesive strength of the oriented polypropylene employed during testing.

Introduction

Future structural applications will involve competition among aluminum, steel and high-strength polymeric materials and composites. In particular, the development of new *in-situ*-fibrillized polymers using techniques such as roll-drawing and hydrostatic extrusion can produce materials with specific strengths and moduli values comparable to metals, and far above the mechanical property levels of present-day engineering plastics. Roll-drawn or uniaxially oriented polypropylene (OPP) with yield strengths up to 500 MPa, and moduli up to 20 GPa has been produced at the University of Toronto (Ref. 1). The electromagnetic induction and ultrasonic bonding of OPP to aluminum has been examined in detail (Refs. 2, 3). These high-strength thermoplastics do not require incorporation of second-phase constituents (such as glass or carbon fibers, or Kevlar) and can be produced on rolling equipment, which has similar characteristics to that employed in the manufacture of steel sheet.

Although ordered thermoplastic laminates (OTLs) or reinforced thermoplastic laminates (RTLs) will compete with metals, the synergistic use of metals and plastics in combination is the likely line of approach in future manufacturing applications. As a result, there will be critical demand for metal/polymer joining technology. The use of adhesive bonding will grow markedly important, but this will require solutions to fundamental problem areas, including health hazards of adhesives used in the workplace and the development of a data bank concerning joint mechanical properties and durability in a range of environments. Also, there will be an increasing demand for alternative joining methodologies to adhesive joining, in particular those involving fusion bonding.

This paper reviews the research presently being carried on at the University of Toronto on joining of polymers to aluminum. The subject of polymer/metal joining encompasses a diverse range of aspects, e.g., the factors controlling adhesion, adhesive chemistry, polymer and metal surface treatments, and the factors affecting initial joint strength and bond line durability in high-temperature/high-humidity environments. The approach taken in this paper involves a brief review of adhesion and adhesives, an analysis of the present status *vis-a-vis* polymer/aluminum and polymer/steel adhesive joining operations and finally the ongoing fusion joining research thrust being followed at the University of Toronto.

KEY WORDS

Polymer/Metal Joints
Polymer Aluminum
Polymer/Steel Joints
Electromagnetic Induction
Ultrasonic Welding
Adhesion Mechanisms
Oriented Polypropylene
Lap-Shear Strengths
Tie-Layers
Fusion Bonding

Adhesion Mechanisms

The thermodynamic work of adhesion, Ø, when an adherend and adhesive are employed during polymer joining is given as,

$$Ø = \Delta_1 + \Delta_2 - \Delta_{12} \quad (1)$$

where, Δ_1 is the surface energy of the adherend, Δ_2 is the surface energy of the adhesive, and Δ_{12} is the adherend/adhesive surface energy.

For solids having different surface energies Δ_x and Δ_y, the work of adhesion depends on,

$$Ø = 2\mu\sqrt{\Delta_x \cdot \Delta_y} \quad (2)$$

where, μ is a constant. Although this equation is generally followed in polymer systems, the strength of polymer/polymer joints generally exceeds the thermodynamic work of adhesion be-

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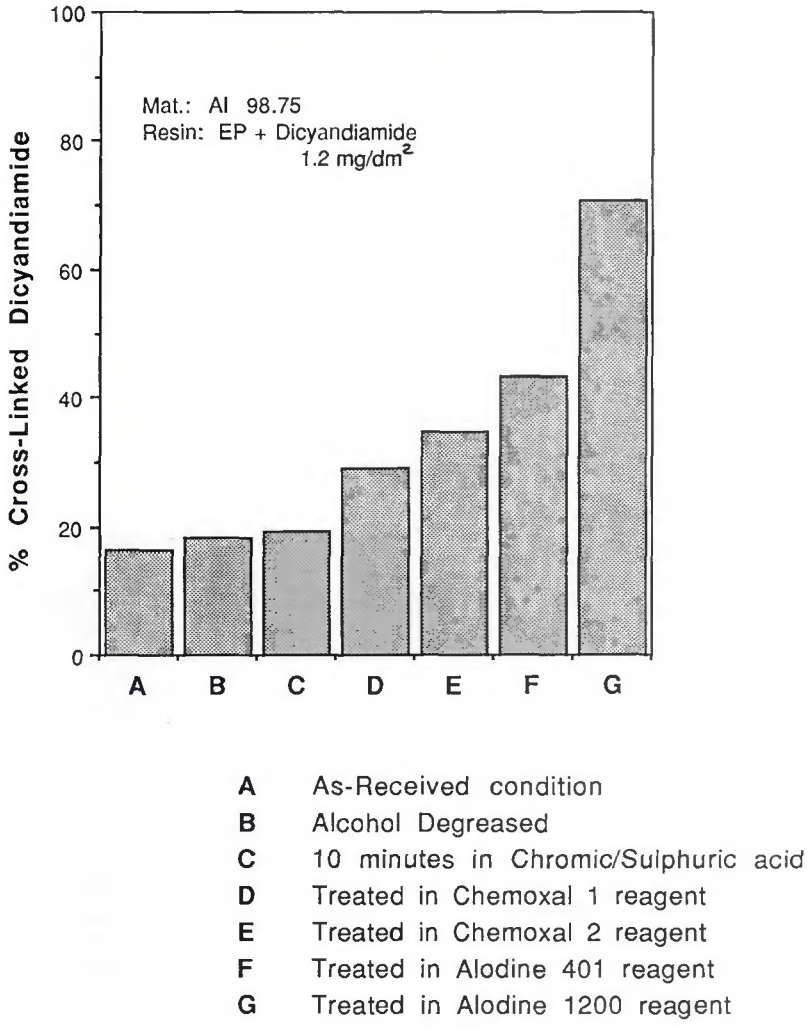

Fig. 1 — Cross-linking density in a thin epoxide coating cured on aluminum after different surface treatments. After Brockman (Ref. 14).

cause polymer chain diffusion (reptation) across the interface produces chain entanglements (Ref. 4). In polymer/metal bonding, there is no chain entanglement effect and final joint strength depends on a number of factors, namely chemical bonding, mechanical interlocking, electrostatic attraction between the metal and the polymer substrates, and on the possible formation of weak boundary layers in the region immediately adjacent to the joint interface. These different factors are summarized below.

Electrostatic/Electronic Theory

When a polymer is in intimate contact with a metal there is a marked charge separation so that an electrical double layer is formed and the joint is strengthened since work has to be applied to separate the two layers of this capacitor (Refs. 5, 6). The contribution of this electrostatic charge force to intrinsic adhesion has been shown to be quite small compared to the contribution from van der Waal's forces (for a system involving adhesion of rubber to a glass interface).

Chemical Bonding

In this case, the adherence of the two substrates depends on the formation of secondary bonds (van der Waal's forces, hydrogen bond formation) and primary bonds (ionic, covalent, metallic bonds and acid base donor-acceptor interactions). Impurities (particularly oxygen) on polymer or on metal surfaces have a strong influence on bond line strength. For example, if polyethylene containing anti-oxidants is used as a hot-melt adhesive, this adheres poorly to a steel surface. Also, ultraclean metal surfaces prepared in vacuum exhibit very poor adhesive properties (Ref. 7). It follows that satisfactory adhesive bonding essentially depends on the application of polymer or metal conditioning treatments, which are made prior to joining. Polymer surfaces can be acid-etched (in different combinations of sulfuric, phosphoric and chromic acid) or plasma or corona arc treated to produce reactive (polar) groups that promote chemical bonding during joining operations. Oxide on the metal surface is particularly important since moisture absorption leads to the formation of hydroxyl groups. These hydroxyl groups on the metal surface can react with polar groups in the adhesive, *e.g.*,

$$\mathrm{MOH + HXR \rightarrow MO \ldots\ldots HXR} \qquad (3)$$
$$\qquad\qquad\qquad\qquad\qquad\quad |$$
$$\qquad\qquad\qquad\qquad\qquad\quad \mathrm{H}$$

where, M is the metal, H is hydrogen, X is an atom of oxygen, nitrogen or chlorine, and R is a polar group. When an adhesive comprising of polyethylene and acrylic acid is used for aluminum bonding, it has been indicated that the optimum concentration of polar groups in the adhesive is around 0.5 to 2 wt-% (Ref. 8). The number of polar groups in a 50-µm-thick film of polyethylene grafted with 1% acrylic acid is 40/100 Å^2. Since the population of –OH groups on a metal surface is around 1 to 50/100 Å^2, it is apparent that optimum strength occurs when the number of available polar groups in the adhesive and the number of available –OH groups on the aluminum surface are similar.

Mechanical Interlocking

When a liquid adhesive is employed, mechanical treatments that increase the surface roughness of the metal produce higher joint strengths. For example, Jennings (Ref. 9) showed that sand blasting produced higher butt joint tensile strength values than grinding when 6061 aluminum and AISI 304 stainless steel sheets were joined using a bisphenol A/polyamide adhesive. This improvement in butt joint strength possibly results from surface cleaning, increased contact area for wetting by the liquid (Ref. 5) and enhancement of energy dissipative mechanisms in the adhesive when the joint is peeled apart (Refs. 10, 11). Microfibrous structures, *e.g.*, produced on copper as a result of wet oxidation, or by electrodeposition of zinc, facilitate mechanical interlocking since highly mobile, low molecular weight fractions in the polymer penetrate the interstices in the microfibrous structure. These microfibrous structures on metal surfaces are characterized by acicular projections only a few microns in length and produce strength concentrations

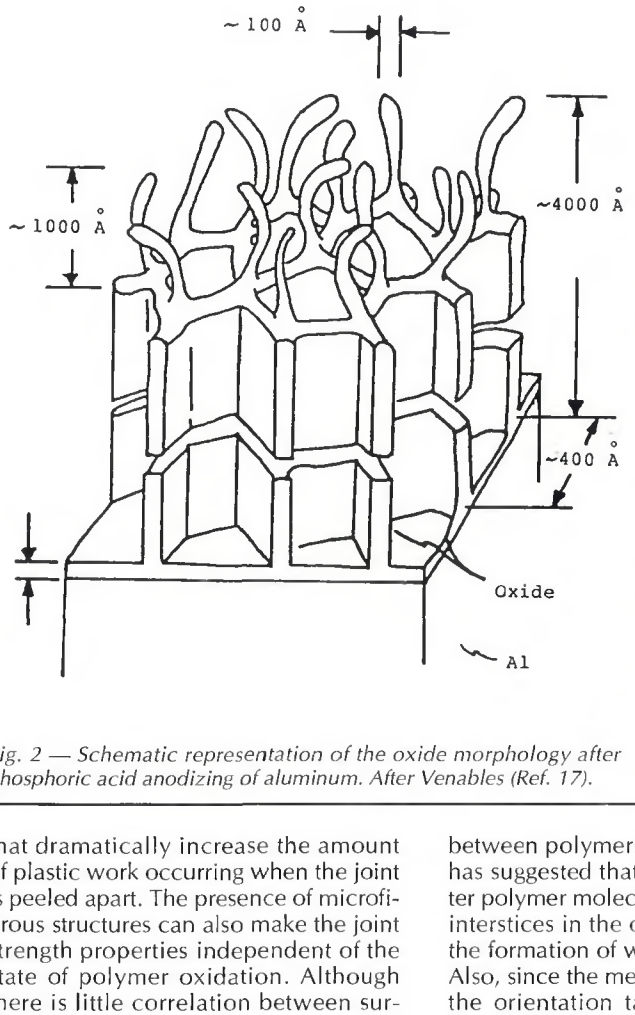

Fig. 2 — Schematic representation of the oxide morphology after phosphoric acid anodizing of aluminum. After Venables (Ref. 17).

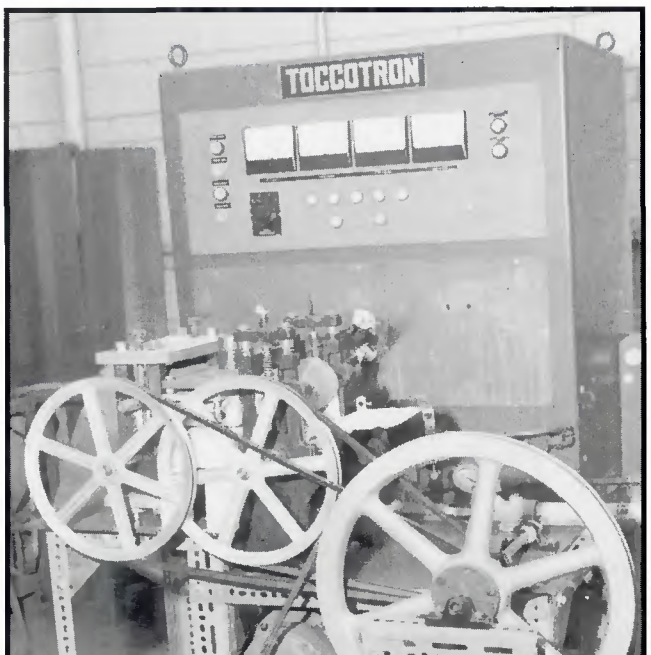

Fig. 3 — Photograph showing the complete induction bonding system including the 450-kHz unit.

that dramatically increase the amount of plastic work occurring when the joint is peeled apart. The presence of microfibrous structures can also make the joint strength properties independent of the state of polymer oxidation. Although there is little correlation between surface macroroughness and joint durability in harsh (high-temperature/high-humidity) environments, it has been suggested that mechanical interlocking on a microscale plays a major role in improving interface durability in the case of anodized aluminum and titanium alloys (Ref. 12). This aspect will be addressed in detail later in this paper.

Weak Boundary Layers

Bikerman (Ref. 13) suggested that failure at the joint interface is improbable and that the presence of a weak boundary layer promotes failure adjacent to, but not actually along the interface. It is well documented that the diffusion of lower molecular weight fractions to the outer polymer surface can lead to the formation of weak surface layers. In fact, this is the underlying principle when addition of short-chain amphipathic molecules are made to prevent adhesion between polymers. Brockman (Ref. 14) has suggested that 20- to 100-Å-diameter polymer molecules can penetrate the interstices in the oxide film and lead to the formation of weak boundary layers. Also, since the metal surface determines the orientation taken up by polymer molecules during absorption, the molecular structure of the adhesive, particularly the degree of polymerization is affected — Fig. 1. For example, in epoxy-bonded steel, the amount of chemical attack on the adhesive film depends on the type of steel surface used, e.g., on grit-blasted steel no attack occurs, while on ground and degreased steel sheet there is significant attack.

Adhesive Bonding of Metals

Aluminum

Structural adhesives are materials which, when cured, possess high modulus and strength, and effectively transmit load across the joint. Aluminum is generally adhesively bonded using thermosetting resins such as epoxies, acrylics, phenolics and temperature-resistant polyimides. These resins harden by a chemical reaction, namely polymerization or condensation.

Chemical surface treatments such as chromic acid etching produce higher adhesive joint strengths than solvent degreasing and grit blasting (Ref. 15). Commonly applied surface treatments for aluminum involve chromic acid etching, chromic acid anodizing and phosphoric acid anodizing. Anodizing involves suspending the workpiece in an acid bath and applying a positive potential. During anodizing, the surface oxide grows in thickness and has a morphology dependent on the acid type and concentration used, on the current density and voltage applied, and on the time and temperature conditions. The phosphoric acid anodizing treatment is generally considered the most effective in terms of joint durability in harsh environments, based on Boeing wedge test results. This particular anodizing treatment depends on the anodic reaction (Ref. 16),

$$Al + 3H_2PO_4^- \rightarrow Al(H_2PO_4) + 3e^- \quad (4)$$

$$2\,Al(H_2PO_4)_3 \leftrightarrow 2AlPO_4 + 4H_3PO_4 \quad (5)$$

Using a variety of surface analysis techniques (extended resolution scan-

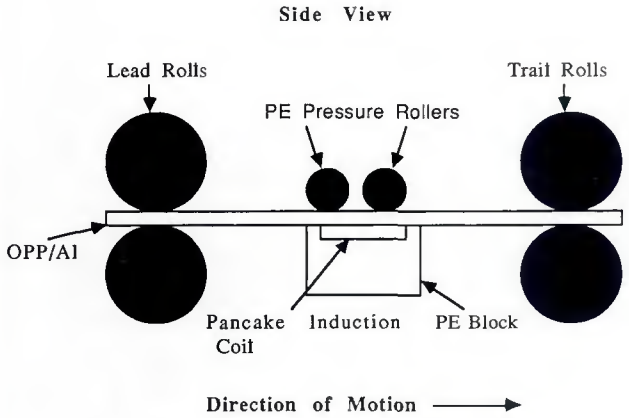

Fig. 4 — Schematic diagram of the continuous electromagnetic induction apparatus.

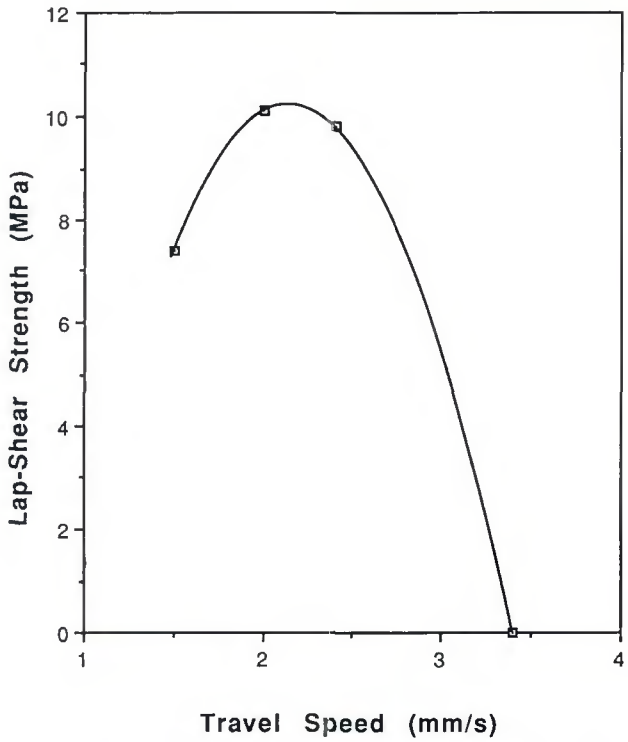

Fig. 5 — Effect of travel speed on the lap shear strength of OPP/aluminum joints produced using electromagnetic induction bonding.

ning electron microscopy, x-ray photoelectron spectroscopy, ellipsometry), Venables (Ref. 17) showed that the oxide morphology after phosphoric acid anodizing was the microfibrous structure that is illustrated in Fig. 2. The long-term durability of phosphoric acid anodized aluminum has been evaluated using surface behavior diagrams (Ref. 18). Davis (Ref. 18) concluded that the adhesive joint durability depended on a multistage hydration process, *i.e.*, involving the reversible adsorption of water by Al PO_4 to form [Al $PO_4 \cdot \frac{1}{2}H_2O$], followed by slow dissolution of the phosphate species exposing the underlying oxide, and finally, hydration of the Al_2O_3 to form boehemite (Al OOH) and then bayerite ($Al(OH)_3$). This change in oxide morphology is particularly detrimental since the boehemite adheres poorly to the aluminum substrate. This assessment of oxide chemistry changes during moisture attack led to the development and application of a hydration inhibitor, nitrolotris methylene phosphonic acid (NTMP), which has been applied to anodized aluminum surfaces and markedly improves bond line durability in adhesively bonded aluminum.

Steels

The initial joint strength in adhesive bonding of steel largely results from chemical bonding, *e.g.*, with the formation of alcolates (with epoxies), and phenolates (with phenolic adhesives). As pointed out earlier, the amount of polymerization in the adhesive near the joint interface depends on the type of steel considered, and in the case of coated steels there are problems (Ref. 7). Acrylic adhesives that set in 1.5 to 2 min on cold-rolled steels, and in 7 to 10 min on electrogalvanized steel, will not cure on hot-dipped galvanized steels. Also, the strength of acrylic-bonded galvanized steels is 9 MN m^{-2} compared to 22 to 42 MN m^{-2} on uncoated steels. The coarse-grained structure of the galvanized layer appears to be the critical feature affecting final joint strength, and successful adhesive bonding depends on mechanical or chemical treatments that remove or alter the outer surface of the galvanizing coating. This explains the higher joint strengths produced after detergent cleaning treatments, after grinding and degreasing and after phosphating. The strength of adhesive joints in galvanized steel can be increased from 6 MN m^{-2} after degreasing, to 15 MN m^{-2} after grinding, and to 28 MN m^{-2} after sand blasting.

Conversion coatings for adhesive bonding of steel usually involve phosphating, and this is usually applied after some abrasive treatment. Although the initial joint strength is not increased by phosphating (it can in fact decrease), the long-term mechanical properties (durability) of the joint in high-temperature/high-humidity environments are unproved if a microfibrous structure is produced during phosphating (Ref. 19). When microporous zinc coatings are employed, bond line failure occurs cohesively within the adhesive layer itself.

Electrogalvanized steel is increasingly in demand and there is a dearth of information concerning basic surface properties, and their variability in product manufactured by different plant-processing routes, *e.g.*, the orientation and crystallite sizes of zinc, surface topography, oxide layer thickness, minor alloy element entrapment, contamination, and so on (Ref. 20). This information is critically needed if optimum mechanical properties are to be achieved in adhesive joints.

In the adhesive bonding of stainless steels anodizing with a range of chemicals (sulfuric acid/chromic acid/phosphoric acid) also produces improved adhesive properties (Ref. 21). The best results are produced using a chromic acid/sulfuric acid anodizing treatment. No detailed information is available concerning the surface character produced using this etching treatment but it is likely that the optimum anodizing treatments are those which form a microfibrous chromium oxide layer on the stainless steel surface.

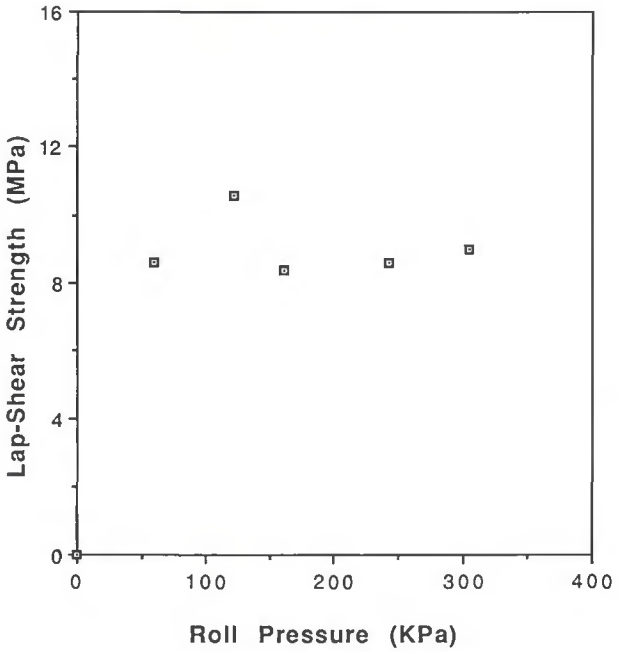

Fig. 6 — Effect of roll pressure on the lap shear strength of OPP/aluminum joints produced using electromagnetic induction bonding.

Fig. 7 — Transmitted, cross-polarized light photomicrograph of an induction weld made with a 450-kHz unit at a speed of 2.5 mm/s and a roll pressure of 120 kPa. A — Formerly Al (that has been dissolved); B — fusion zone consisting of a first transcrystalline region adjacent to the Al interface, a region of randomly oriented spherullites and a second transcrystalline region next to the partially disoriented OPP; C — heat-affected zone consisting partially disoriented OPP; D — fully oriented OPP. (62.5X).

Fusion Joining of Polymers to Metal

Electromagnetic Induction Bonding of Oriented Polypropylene to Aluminum

The approach in electromagnetic induction bonding for joining high-strength, roll-drawn polypropylene (OPP) to aluminum involves phosphoric acid anodizing of the aluminum sheet, the use of high-frequency induction to heat the aluminum/OPP interface and the application of pressure to keep the sheets together as they pass through a set of drive rolls (Ref. 22). The welding equipment (Figs. 3 and 4) consisted of: 1) a 107 X 80-mm (4.2 X 3.1-in.) four-turn, rectangular pancake inductor mounted in a polyethylene block and covered with 0.8-mm (0.03-in.) thick PTFE for isolation purposes; 2) a roll-train controlled by a variable DC motor, which allows the polymer and aluminum sheets to be driven through at speeds up to 2.5 mm/s; and 3) a pressure-loading setup comprising two polyethylene rollers located directly above the induction coil.

The welding unit was a TOCCO 450 kHz device with an output of 25 kW. During testing, the power output of the induction coil was maintained constant (by matching the work coil with the tank circuit). 0.4-mm (0.016-in.) thick 2025-T3 aluminum sheet was used throughout. In tests examining the influence of travel speed, this was varied from 0 to 2.5 mm/s (6 in./min) at a constant applied pressure of 120 kPa (17.4 psi). The influence of roll pressure on lap shear strength was evaluated by varying this parameter from 0 to 300 kPa (43.5 psi), while the travel speed was maintained at 2 mm/s (4.7 in./min).

Figures 5 and 6 show the effects of travel speed and roll pressure on the lap shear strength of OPP/aluminum joints. Optimum lap shear strength (10 ± 1.6 MPa) occurred at a travel speed of 2.0 mm/s and roll pressure had little influence on lap shear strength when it exceeded 50 kPa (7.3 psi). At low travel speeds, the OPP/aluminum interface overheated (producing chain scission and porosity formation), while at high travel speeds there was inadequate fusion of the OPP material. The optimum peel strength of OPP/aluminum joints was 2.9 ±0.6 kN/m when using a travel speed of 2.0 mm/s and a loading pressure of 120 kPa.

The mechanical properties of electromagnetic induction bonded OPP/aluminum exceeded those found when using a two-part, rubber toughened epoxy adhesive (the lap shear strength was 7.0 ±1.9 MPa in this case).

Microtomed cross-sections of test welds indicated the existence of 10-µm-diameter transcrystalline spherulitic morphology at the boundary between the aluminum and the polymer melt, and 1- to 5-µm-diameter transcrystalline spherulitic structures at the boundary between the polymer fusion zone and the heat-affected zone of the oriented polypropylene material — Fig. 7. These transcrystalline spherulitic regions have already been observed when polymer melts are cast against high energy surfaces such as aluminum, and other polymers such as polyethylene terephthalate (Refs. 23, 24)). The nucleation of transcrystalline spherulitic structures depends on the subtle changes in chemistry of the nucleating surface, mechanical stress and cooling rate. The trancrystalline spherulitic regions illustrated in Fig. 7 have mechanical properties quite different from the bulk polymer (higher storage and loss moduli values), higher density and surface energy, and consequently, markedly improved adhesion characteristics (Ref. 25).

Ultrasonic Bonding of Oriented Polypropylene to Aluminum.

In conventional ultrasonic joining, an energy director is employed to promote fusion at the bond line region. This approach has inherent problems in that the energy director has to be specially molded on the polymer substrate. Another line of approach has been taken

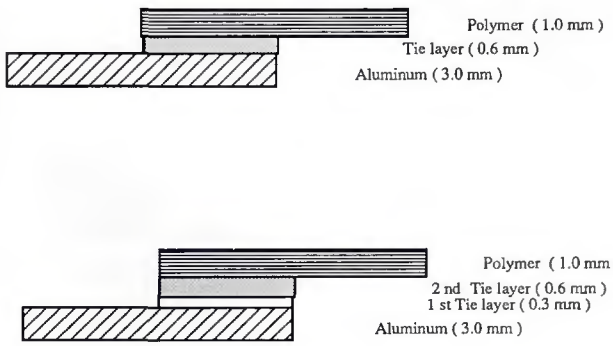

Fig. 8 — Schematic of single and double tie-layer assemblies used in ultrasonic bonding.

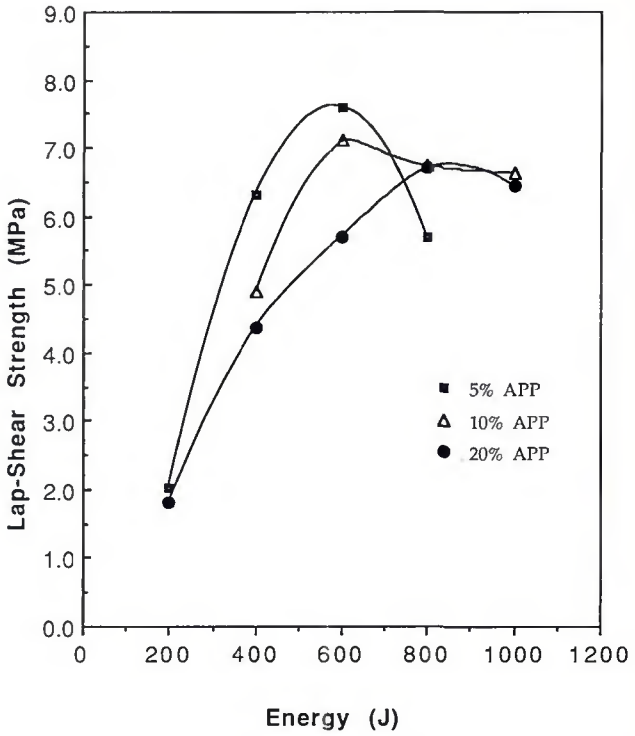

Fig. 9 — Effect of energy input on the lap shear strength of single tie-layer ultrasonic bonds.

at the University of Toronto, *viz.*, the use of tie-layers, which preferentially fuse at the bond line region during ultrasonic bonding. This approach has been successfully employed for the joining of high-strength polypropylene (Ref. 2), and the present work extends the tie-layer approach to metal/polymer joining.

The materials employed during testing was comprised of 3-mm (0.12-in.) thick 6061-T6 aluminum alloy sheet and 1-mm (0.04-in.) thick oriented polypropylene (OPP) produced by roll-drawing (the detailed aspects of roll-drawing are presented in Ref. 1. The oriented polypropylene had a draw ratio between 11 and 12 and a tensile strength of 300 MPa in the direction of rolling. A detailed flow diagram for aluminum pretreatment is given in Table 1. Different tie-layer formulations were examined during ultrasonic bonding, *viz.*, polypropylene (PP, Himont, SV-802) and polypropylene containing 5, 10 and 20 wt-% of amorphous polypropylene (APP). All tie-layers were produced by blending in a Gelimat K-mixer at a speed of 3500 rpm, discharging them from the mixer at 165°C (1202°F) and, through a series of steps, pressing them to thicknesses of and 0.6 ± 0.03 mm (0.02 in. ± 0.001 in.) between Mylar sheets at 185°C (365°F) in a Wabash press.

The effectiveness of the anodizing treatment was evaluated using lap shear testing (ASTM D3163-73 standard). Aluminum sheets were joined together using a polypropylene tie-layer in a hot press. During hotpressing, the conditions were 12.2-ton load for 20 min at a temperature of 185°C. The overlap length was 25.4 mm (1 in.), and during testing all specimens failed cohesively in the polymer layer (not interfacially along the polymer/aluminum interface), confirming the adequacy of the aluminum surface conditioning technique employed during experimentation.

Two approaches were employed in ultrasonic bonding, *viz.*, single and doubletie-layer assemblies — Fig. 8. The first approach consisted of using PP + APP as a single-tie layer for ultrasonic bonding. The second approach consisted of hot pressing the PP tie layer on to the aluminum first to give a final thickness of 0.3 ± 0.03 mm, followed by ultrasonic bonding using a PP + APP second-tie layer. Ultrasonic bonding was carried out using a Sonics & Materials device, which had a maximum output power of 1700 W, and at least five samples were tested to evaluate the shear strength of the welds. Figures 9 and 10 show the lap shear strengths of ultrasonic welds produced using the single and double tie-layer approaches. The amplitude of vibration and the applied load during ultrasonic welding were maintained constant at 80 µm and 0.207 MPa, respectively.

The highest lap shear strength values were produced using a tie-layer formulation containing 5% APP and an energy input of 600 J for both the approaches. All failures in ultrasonic joints exhibiting the highest lap shear strength levels occurred cohesively in the oriented polypropylene sheet. The highest lap shear strength values in the double-tie-layer assembly (11 MPa) equaled the interfibrillar strength in the oriented polypropylene, *i.e.*, the stress required to pull the oriented fibrillar structure apart. The lap shear strength (Ref. 8 MPa) of the single tie-layer assembly was less than that in the double tie-layer case. This difference may have resulted from the change in geometry of the lap shear test specimens, *i.e.*, increasing the width of the adhesive (by using two tie-layers) decreases the stress concentration at the overlap edges, and hence increases the lap shear strength of the joint (Ref. 26).

This research program is ongoing and future work will examine bond line durability in tests carried out in a range of high-temperature/high-humidity environments, the effects of chemically modified tie-layer materials, and detailed chemistry of the bond line region.

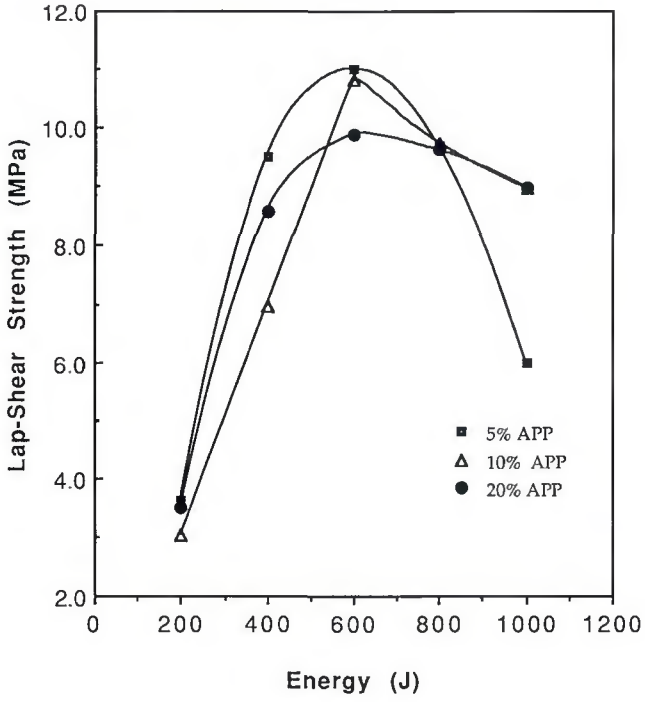

Fig. 10 — Effect of energy input on the lap shear strength of double tie-layer ultrasonic bonds.

Conclusions

A considerable amount of research has been carried out on adhesive bonding and much of this knowledge has been applied to aluminum joining. More is known concerning adhesive bonding of aluminum than in the case of steel. In particular, electrogalvanized steels are increasingly in demand and a detailed evaluation of fundamental surface properties is needed to optimize polymer/metal bonding.

Both electromagnetic and ultrasonic fusion joining techniques were applied to polymer/metal bonding. The lap shear strength of electromagnetic induction-bonded joints between oriented polypropylene and aluminum had shear strengths of 10 ± 1.6 MPa and were produced at an optimum travel speed of 2.0 mm/s. The lap shear strength of these fusion welds was higher than those produced by adhesive joining using acrylic and epoxy adhesives.

In the case of ultrasonic bonding, the approach depended on the use of specially formulated tie-layers, which preferentially fused at the OPP/aluminum interface. The highest lap shear strengths (11 MPa) were produced using tie-layers comprising 5% amorphous polypropylene in a double tie-layer assembly. Joint failure during lap shear testing always occurred away from the joint interface, and bond line strength exceeded the interfibrillar cohesive strength of the oriented polypropylene material. Al-

Table 1 — Flow Diagram for Aluminum Pretreatment

solvent wiping with 1,1,1 - trichloroethane

↓

alkaline degreasing and etching in aq. sol. of 250 g/L of NaOH at room temperature for 10 min.

↓

rinsing in cold water

↓

de-smutting in an aq. sol. of 50% by vol. of HNO_3

↓

rinsing in cold water

↓

anodizing in a stainless steel bath containing an aq. sol. of 10% by weight of 85% phosphoric acid, maintained at a temperature of 19 to 21°C for 25 min. at 25 V

↓

rinsing in cold water

↓

priming with an aq. sol. of NTMP

↓

drying and dessicating

though the present paper discusses only ultrasonic joining, the novel technique of placing thin custom formulated polymer films at the interface offers a great potential for joining polymers to a range of substrates such as metals, composites, ceramics and also to dissimilar polymers using a variety of joining techniques.

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Nitrogen in Arc Welding — A Review

By IIW Commission II

In 1983, Commission II of the International Institute of Welding (IIW) initiated an effort to review and examine the role of nitrogen in steel weld metals. The objective was to compile in one source, for future reference, the available information on how nitrogen enters weld metals produced by various arc welding processes, what forms it takes in these welds, and how it affects weld metal properties.

This bulletin contains 13 reports and several hundred references related to Nitrogen in Weld Metals that has been prepared as a review to show the importance nitrogen has in determining weld metal properties.

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