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# Structural design and bonding strength evaluation of Al/epoxy resin joint via interpenetrating phase layer



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#### ABSTRACT

The effect of the volume fraction of resin ( $V_r$ ) on the bonding strength of Al/epoxy resin joints via interpenetrating phase layer (IPL) was investigated. The tensile strength reached maximum to be 38.3 MPa when  $V_r$  was 85%. Fracture occurred at either epoxy resin/IPL interface or Al/IPL interface depending on  $V_r$  ( $V_r \leq 85\%$ ; epoxy resin/IPL interface,  $V_r \geq 85\%$ ; Al/IPL interface). Fractography revealed that the separation and the fracture of the epoxy resin occurred on the epoxy resin/IPL interface, and the separation and the fracture of Al occurred on the Al/IPL interface. These results can be explained by the strength of each interface estimated from rule of mixture. The effect of grading  $V_r$  on the bonding strength was also investigated. The tensile strength of joint with the functionally graded IPL structure was higher than that of joints with the homogenous IPL structures.

#### 1. Introduction

In view of reducing  $CO_2$  emission and improving the fuel efficiency, weight reduction of vehicle bodies has been required. "Multi-material structure" has been focused on, where the most suitable material (e.g. steel, light metal, carbon-fiber-reinforced plastic (CFRP), etc.) is used for designing each component of the vehicles. Goede et al. (2009) reviewed the motivation and objectives for the multi-material structure. The vehicles can be designed considering the balance between high strength and low weight. On the other hand, technologies to join dissimilar materials like metals and resins should be established.

Amancio-Filho and dos Santos (2009) summarized various methods of joining metals and resins. Adhesives or mechanical fastening components (e.g., bolts and nuts) are widely used practically to join metals and resins. Adhesives can be applied to almost all materials and achieve strong bonding due to equally distributed stress on the entire surface. However, their life span is unclear, and Venables (1984) reported that adhesives degrade easily when used in high-temperature and high humidity environments. Moreover, adhesives contain volatile organic compounds (VOCs), which may damage the environment and human health. Guo et al. (2011) measured the total emissions of the VOCs from adhesives and found that the time-dependence of the emissions was expressed in the exponential function. The VOCs can damage the environment and human health. For example, Guo et al. (2004) showed that the VOCs had an effect on the life time cancer risks. When mechanical fastening components are used, highly reliable joints can be achieved, and the assembly and disassembly of composites are easier. Boothroyd and Alting (1992) summarized the product design for the assembly and disassembly. However, as the number of components increases, the total weight of the end-product also increases. In addition, stress concentrates on the joint, which is the starting point for the rupture. Amancio-Filho and dos Santos (2009) and Kah et al. (2014) stated stress concentrates on the joint, which is the starting point for the rupture. Therefore, a direct method for joining metal and resin is required.

One of the direct joining methods is the mechanical interlocking utilizing anchor effect. Metal and resin interlock each other by infiltrating resin into dimples formed on the metal surface. The surface structure on the metal is important to obtain high bonding strength. Many potential methods for modifying the metal surface are reported, e.g. laser irradiation, chemical etching, sandblasting and so on. Nielsen et al. (2010) and Kurakake et al. (2013) modified the surface structure of a stainless steel and an aluminum alloy, respectively, by laser irradiation. In addition, Roesner et al. (2011) and Amend et al. (2013) reported the thermal joining between a steel or an aluminum alloy and resins after modifying the metal surface by laser irradiation. Kim et al. (2010) coated a photoresist film in a pattern on the surface of a steel plate and chemically etched the uncoated area by Nital solution. Kimura et al. (2016) formed many pores with nanometer size on the surface of an aluminum alloy by chemical processing. Harris and Beevers (1999) treated a mild steel and an aluminum alloy by grit blasting. Including these researches, there have been many studies on

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the shear strength at the interface in the case of the anchor bonding. However, the tensile strength perpendicular to the interface has not been widely investigated. In addition, many processes for providing anchor structures have been developed, but few studies have systematically investigated the correlation between the anchor structure and the bonding strength. Kleffel and Drummer (2017) investigated the correlation between the tensile strength and the surface roughness using the single rib joints, but the tensile strength could not be understood by only the roughness parameter.

Recently, our group (Suzuki et al. (2018)) have proposed a novel concept of the anchor bonding between metal and resin via "interpenetrating phase layer (IPL)". Open porous layer is formed on the metal surface, resin infiltrates into the layer and the metal and the resin interlock three-dimensionally. High bonding strength can be achieved in all directions due to the three-dimensional interlocking of the IPL. In our previous study, it was revealed that IPL thickness should be appropriately controlled for high anchor effect and stress relaxation. For this bonding method, open porous structure reflects the IPL structure. For example, porosity in the porous layer corresponds to the volume fraction of resin in the IPL ( $V_r$ ). In this study, by controlling the porosity in the porous layer, we investigated the effect of  $V_r$  on the tensile bonding strength of Al/epoxy resin joints.

#### 2. Materials and methods

Porous Al layer was obtained by the spacer method using sodium chloride (NaCl), which was developed by Zhao and Sun (2001). This method permits controlling the porosity and fabricating an open porous structure suitable for resin infiltration. First, in order to assess the connectivity of pores and the controllability of the porosity, porous Al samples were fabricated using various volume fractions of NaCl. Al powder (purity: 99.99%, average particle size: ~20 µm) and NaCl powder (purity: 95%, particle size: 330-430 µm) were used. The powders were blended manually so that the volume fractions of NaCl were 30, 40, 50, 60, 70, 80 and 90%. The Al/NaCl mixture was placed into a graphite mold (inner diameter: 10 mm, outer diameter: 20 mm, height: 50 mm) and treated by electric current sintering in vacuum ( $\sim$  30 Pa) while 30 MPa of pressure was applied. Temperature was raised at a rate of 0.5 °C/s and held at 570 °C for 600 s, followed by furnace cooling. The maximum voltage and current during the sintering were  $\sim 2.8 \text{ V}$ and  $\sim$  230 A, respectively. The sintered samples were then kept under water for more than  $8.64 \times 10^4$  s so that NaCl leached away to obtain open porous structures. Finally, the samples were dried in an electric furnace at 150 °C for more than  $3.6 \times 10^3$  s. The removal ratio of NaCl and the porosity were calculated by following equations.

Removal ratio of NaCl = 
$$\frac{m_{\text{sin tered}} - m_{\text{leached}}}{m_{NaCl}}$$
, (1)

Porosity = 
$$1 - \frac{\rho_{\text{sample}}}{\rho_{\text{Al}}} \left( \rho_{\text{sample}} = \frac{4m_{\text{leached}}}{\pi d^2 h} \right),$$
 (2)

where,  $m_{\text{sintered}}$  and  $m_{\text{leached}}$  are mass of the samples after sintering and NaCl removal,  $m_{\text{NaCl}}$  is mass of NaCl in the powder mixture,  $\rho_{\text{sample}}$  is the bulk density of the sample after NaCl removal,  $\rho_{\text{Al}}$  is the true density of Al (2.7 g cm<sup>-3</sup>), *d* and *h* are diameter and height of the cylindrical sample (d = 10 mm, h = 10 mm).

Next, Al/epoxy resin joints were fabricated in order to evaluate the tensile bonding strength. The powders were blended manually so that the volume fractions of NaCl were 70, 75, 80, 85 and 90%. Al powder was compacted in the cylindrical graphite mold (inner diameter: 20 mm, outer diameter: 40 mm, height: 50 mm) at 30 MPa. Then, the Al/NaCl mixture was placed on top of the Al green compact and treated by electric current sintering at the same condition as above. The end surface of the (Al/NaCl) layer of the sintered samples was ground using a lathe so that the thickness of the IPL was 2 mm. NaCl leached away and the samples were dried by the method as mentioned above.

#### (a) Al/epoxy resin joint via IPL



**Fig. 1.** The size of the tensile test specimens of (a) the Al/epoxy resin joint via IPL, (b) the sintered Al and (c) the epoxy resin.



Fig. 2. Change in the removal ratio of NaCl as a function of the volume fraction of NaCl in the raw powder mixture.

Bisphenol A-type epoxy resin was blended with a modified aliphatic polyamine curing agent, and infiltrated into the porous layer under reduced pressure ( $\sim$ 4 kPa). The sintered Al with the porous layer was placed in a container within an evacuated bell jar for 600 s. The epoxy

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