X-ray Stress Measurement for Ni-Al System
Intermetallic Compound Prepared by SHS- method

*Takayuki MUROTANI  *Juwen HE **Toshihiko SASAKI and ***Hajime HIROSE

*Graduate student of Kanazawa University, Kakumamachi Kanazawa Ishikawa 920-1192, Japan
**Dept. of Material Science and Engineering, Kanazawa University, Kakumamachi Kanazawa Ishikawa 920-1192, Japan
***Kinjo University research center, Kasamamachi 1200, Matto, Ishikawa 924-8511, Japan

ABSTRACT
An X-ray diffraction technique was used to measure the residual stress in the surface of a specimen containing a coating of a Ni$_3$Al intermetallic compound produced by the SHS (Self-propagation High temperature Synthesis) method. The Ni$_3$Al was using the coating material for spherical carbon cast iron substrate and austenite stainless steel substrate. Differences between the coefficient of thermal expansion of the coating and substrate were confirmed to be the source of compression stress in the coating.

INTRODUCTION
The recent application of composite material that is low in cost and has an excellent coating material with heatproof, wear-resistant corrosion, and surface modifications is increasing. However, the self-propagation high-temperature synthesis (SHS) method is a technique that uses the chemical reactions between the elements that compose the intermetallic compound. In this method the powder mixture is heated and is able to generate the combustion reaction of the powder elements by keeping the heating temperature under the melting point of the given compound. In this study, our purpose is to coat the intermetallic compound on spherical carbon cast iron and on austenite stainless steel substrate by using the SHS method and to investigate the stress condition inside the coating layer.

The thermal stress inside the material where heat and bond exist is considered to influence the material strength of the coating layer. After where producing the foundation material, the residual stress distribution inside the Ni$_3$Al coating layer was measured for each specimen.

SPECIMEN
We produced the coating layer by using the combustion synthesis reaction of the powder compact. The powder was composed of elements in the atomic ratio Ni-25at%Al from Ni powder (purity 99.9wt%, mean grain size 5 μm) and Al powder (purity 99.9wt%, mean grain size 3 μm). The mixed powder was compacted to diameter=10mm, and thickness=0.187mm at 400MPa by using the metal dice. The chemical compositions of the substrate austenite stainless steel (Japanese Industrial Standard: JIS-SUS304) and spherical graphite cast iron (JIS-FCD700) are shown Table 1. The micrograph of the cross section of the powder compact is shown in Figure 1. Since the part that is forming 10~20 μm spherical clusters in this micrograph, surrounds Ni, is Al, and we are able to confirm that it
is mixing evenly macroscopically. The specimen was fixed among the ceramic dice of the hot press device, as shown in Figure 2. The vacuum in the furnace was $660 \times 10^{-3}$ Pa. While pressing mechanically with 20MPa from the vertical direction of the interface, the inside temperature of the furnace was brought to 1023K in approximately 2.4ks. Hot press held the furnace constant temperature of 1023K, 0.6ksec. The cross section in the vertical direction of the specimen coated on the SUS304 substrate is shown in Figure 3. The joining quality was fine and no voids were observed in the interface area. The specimen with FCD700 as the substrate exhibited similar conditions.

Fig.1 Micro structure of powder compact.

Fig.2 SHS apparatus.

Fig.3 Micro structures of specimen interface.

Fig.4 Coordinate system of X-ray stress measurement.

Table 1 Chemical property and dimensional of substrate material. Table 2 Coefficient of thermal expansion.$^{[4]}$

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Contents, mass%</th>
<th>Ni$_3$Al</th>
</tr>
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<tbody>
<tr>
<td>SUS304</td>
<td>C:0.07 Si:0.42 Mn:1.20 P:0.028 S:0.022 Ni:8.3 Cr:18.8</td>
<td>$11.9 \times 10^{-6}$ K$^{-1}$</td>
</tr>
<tr>
<td>FCD700</td>
<td>C:3.50 Si:2.56 Mn:0.28 P:0.021 S:0.006 Mg:0.039</td>
<td>$17.8 \times 10^{-6}$ K$^{-1}$</td>
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DISTRIBUTION OF RESIDUAL STRESS

The residual stress distribution measurement used the RIGAKU MSF-2M X-ray stress measurement device with the \( \sin^2 \phi \) technique\(^5\). The measurement conditions are shown in Table 4. The measurement region was at the central part of the surface of the Ni\(_3\)Al coating layer (Figure 4). The optic system was set up in the radial direction \( X_1 \) for \( \sigma_x \). To enable the residual stress distribution measurement in the vertical direction from the surface to joining interface direction, the surface layer was removed one by one by the electrolysis grinding technique using 5% sulfuric acid 95% methanol. The X-ray elastic constant used was calculated using the Kröner\(^6\) model from the stiffness tensor \(^7\) of the Ni\(_3\)Al single crystal.

<table>
<thead>
<tr>
<th>X-ray method</th>
<th>Parallel beam</th>
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<tbody>
<tr>
<td>Characteristic X-ray</td>
<td>V-K ( \alpha )</td>
</tr>
<tr>
<td>Wave length, nm</td>
<td>0.250</td>
</tr>
<tr>
<td>Filter</td>
<td>Ti</td>
</tr>
<tr>
<td>Tube voltage, kV</td>
<td>30</td>
</tr>
<tr>
<td>Tube current, mA</td>
<td>10</td>
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<tr>
<td>Irradiated area, mm(^2)</td>
<td>4\times6</td>
</tr>
<tr>
<td>Diffraction plane, hkl</td>
<td>Ni(_3)Al 220</td>
</tr>
<tr>
<td>Diffraction angle, deg.</td>
<td>163.8</td>
</tr>
<tr>
<td>Peak position</td>
<td>Half value method</td>
</tr>
<tr>
<td>Measurement optics</td>
<td>Iso-inclination method</td>
</tr>
<tr>
<td>X-ray elastic constant ( E/(1+\nu) ), GPa</td>
<td>169.23</td>
</tr>
<tr>
<td>Stiffness(Single crystalline), GPa</td>
<td>( C_{11}=169, C_{44}=121, C_{12}=89 )</td>
</tr>
</tbody>
</table>

EXPERIMENTAL RESULTS AND DISCUSSION
RESIDUAL STRESS DISTRIBUTION IN Ni\(_3\)Al COATING

The 2\( \theta \)-\( \sin^2 \phi \) diagrams of the coating layer are shown in Figures 5(a)\(~(c)\). The 2\( \theta \)-\( \sin^2 \phi \) diagrams other than that of the SUS304 substrate in Figure 5(c) show linear behaviour. However, the 2\( \theta \)-\( \sin^2 \phi \) diagram in Figure 5(c) is curved. The curve of the 2\( \theta \)-\( \sin^2 \phi \) diagram like that for the SUS304 substrate did not exist for the specimen with the FCD 700 substrate. Namely, this curve is not a result of the influence of the texture that forms during crystal growth, i.e., from generation to cooling of Ni\(_3\)Al. Similarly, the influence of the texture was not observed in the diffraction intensity ratios for either specimen, in the X-ray diffraction profiles, which it shows to Figure 6.

The measurement result of the residual stress distribution of joining interface depth direction was shown to Figure 7. The sharp decay of the compression residual stress is observed in the regions near interface. Accordingly, the cause of the curve of the 2\( \theta \)-\( \sin^2 \phi \) diagram of the SUS304 substrate specimen is considered to depend on the existence of a sharp stress gradient of depth direction\(^8\).
The measurement result of the residual stress distribution of the joining interface in the depth direction is shown in Figure 7. The sharp decay of compression residual stress is observed in the regions near the interface. Accordingly, the existence of the curve of the
2θ - sin² φ diagram of the SUS304 substrate specimen is considered to depend on the existence of a sharp stress gradient in the depth direction[8]. In this experiment, the compression residual stress inside the Ni₃Al coating layer for the specimen with the SUS304 substrate was maximum at approximately 60 μm from the joining interface. The curve in our results agrees with a previous report on the maximum stress for different materials joining in the interface area[9]. The coating layer inside the specimen with the substrate of FCD700 was under the residual stress of slight compression. The main cause of these residual stresses is conceived to be the influence of the heat stress that originates from the differences of the mechanical properties such as the coefficients of thermal expansion of the substrate material and coating layer.

CONCLUSIONS

In this experiment, the Ni₃Al intermetallic compound was coated on a spherical carbon cast-iron substrate and austenite stainless steel substrate by using the self-propagation high-temperature synthesis method. X-ray stress measurement was carried out in the coating layer surface. On the basis of our results, the following conclusions were made,

(1) In the specimens of Ni₃Al coated on SUS304 and FCD700 substrates using the SHS method, compressive residual stress exists.
(2) In the residual stress distribution in the depth direction of Ni₃Al on SUS304 substrate, a maximum value is obtained at about 60 μm from the joining interface which is dependent on the stress condition of the material and the type of coating on the substrate.
(3) These results are considered to be influenced by the heat stress caused by the difference between the coefficients of thermal expansion for coating and substrate materials.

REFERENCES