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FORMATION OF THE (γ/α) - INTERPHASE BOUNDARIES IN FeCrNi ALLOYS BY A DIFFUSION BONDING METHOD

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Abstract - Characteristics of the interphase boundary in two-phase (γ/α)-stainless steel couples produced by a solid-solid diffusion method, starting from Fe-30%Cr(α-phase) and Fe-15%Cr-15%Ni(γ-phase) alloys, have been described. Interphase boundaries between a parent γ-phase and a transformed α-phase have been found to be essentially planar and no special structural defects have been detected, excepting a so-called "diffusion-induced twins" grown often in the γ-phase, similar to those found in the diffusion couples of α/β-brass bicrystals by one of the authors and his colleagues/1/. It is found that the transformed α(αn)-phase inherits the crystallographic orientation of the original α(αn)-phase in a similar manner to GRAIN GROWTH. However, if the bonding orientation relation is set to be a near Kurjumov-Sachs relation, NUCLEATION of the phase transformation occurs and the resulting orientation relations become closer to the preferential one. The present results suggest that the critical condition for the occurrence of the nucleation is attributable to the interfacial energy criterion on the (γ/α)-interface and the grain boundary between the αo- and αn-phases.

1 - INTRODUCTION
A model consisting of a single crystal of one phase joined to a single crystal of another phase, i.e. a two-phase bicrystal, is desirable to evaluate the structure of the interface and the role of the interface on mechanical properties. The different methods which provide such bulk bicrystal were reviewed briefly/2/. The present author and his colleagues have developed a solid-solid diffusion bonding method/3,4/, which is founded on a classical diffusion in the solid state, i.e. when two phases which are consecutive in the phase diagram are pressed together and diffused at a high temperature, only one interface between the phases will appear and will move towards the one phase, according to the occurrence and development of a phase transformation.
This method has been first applied to the \((\alpha/\beta)\)-CuZn system/3-5/, then to the \((\alpha/\beta)\)-CuAl/6/, AgZn/7/ and AgMg/8/ systems. Orientation relations (ORs) between the parent \(\alpha\)-phase and the transformed \(\beta\)-phase in almost all of the bicrystals in these systems are found to be the Kurjumov-Sachs (KS) or Nishiya-Wassermann (NW) relations, which are well-known preferential ORs between the fcc- and bcc-phases developed under the diffusional or martensitic phase transformations. The fact clearly suggests that at the early stage of diffusion-bonding treatment a nucleation of the grain must be taken place in order to attain such ORs, generally resulting a formation of grain boundary between the old and new \(\beta\)-phases, which may bring a high interfacial energy state. However, there has been appeared no solid answer based upon the experiment whether this kind of nucleation is prerequisite or not.

The purposes of the present study are: (1) to observe macroscopic structure of the interphase boundary between the \(\gamma\) - and \(\alpha\)-phases in two-phase stainless steel couples produced by the diffusion-bonding method; (2) to clarify the necessary condition of the occurrence of the nucleation.

2 - EXPERIMENTAL

Chemical compositions of the materials used are shown in Table 1. The compositions of Fe are fixed to be about 70\%, and those of Cr and Ni are chosen in order to obtain an appropriate diffusion of the elements between the \(\gamma\) - and \(\alpha\)-phases. Four types of diffusion couples consisting of single crystal (S) and polycrystal (P) of each phase were made, called S/S, P/S, S/P and P/P couples, where the numerator and denominator correspond to the \(\gamma\)- and \(\alpha\)-phases, respectively.

Single crystals in the form of rod (diameter = 10 - 12mm, length = 10cm) of both phases were grown using the Bridgman method in an argon gas atmosphere. Both the single crystals and the polycrystals were cut in plate. The well polished plates were abraded with diamond paste of 1 \(\mu\)m to obtain good contact. Couples were clamped together in a specially designed compressive device (material: SKD61) and heated in a furnace controlled to \(\pm 1K\) in an argon gas atmosphere. Molybdenum plates of 0.3mm thickness were put between a couple and compressive disks in order to suppress diffusion of carbon from the device into the couple. The diffusion temperatures and times used in the present experiments were generally between 1073 to 1528K and 50 to 200h, respectively. After the diffusion treatment, each couple was quenched in water to avoid precipitation of the \(\gamma\)-phase in the \(\alpha\)-phase.

The metallographic features were revealed using an electrolytic etching technique with a solution of oxalic acid (10\%). Crystallographic orientations of the both phases were measured within an accuracy of better than 0.2 degrees using a computer aided Laue back-reflection method/9/. The concentration profile of the diffusion couple was determined on an Electron Probe Microanalyzer (EPMA)

<table>
<thead>
<tr>
<th>Phase</th>
<th>C (wt.%)</th>
<th>Si (wt.%)</th>
<th>Mn (wt.%)</th>
<th>P (wt.%)</th>
<th>S (wt.%)</th>
<th>Cu (wt.%)</th>
<th>Al (wt.%)</th>
<th>Cr (wt.%)</th>
<th>Ni (wt.%)</th>
<th>Fe (wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\gamma)-SS</td>
<td>0.0022</td>
<td>0.012</td>
<td>0.009</td>
<td>0.0014</td>
<td>0.0014</td>
<td>(&lt;0.001)</td>
<td>0.002</td>
<td>14.59</td>
<td>14.54</td>
<td>bal.</td>
</tr>
<tr>
<td>(\alpha)-SS</td>
<td>0.005</td>
<td>0.008</td>
<td>0.001</td>
<td>0.003</td>
<td>0.0012</td>
<td>(&lt;0.001)</td>
<td>(&lt;0.001)</td>
<td>29.56</td>
<td>-</td>
<td>bal.</td>
</tr>
</tbody>
</table>

3 - RESULTS

3.1 Migration of the Interface

Migration of the interphase interface after a diffusion treatment and concentration profiles measured by an EPMA are represented in Fig.1. Figure 1(a) is a representation of concentration profiles of Ni and Cr in a diffusion couple treated at 1518K for 145h. A part of the phase diagram of the FeCrNi alloy system is shown in (b). Where there are two phases of \(\gamma\) and \(\alpha\) with concentrations \(C(\gamma0)\) and \(C(\alpha0)\), respectively, an interface appears by diffusion having limit concentrations \(C(\gamma/\alpha)\) and \(C(\alpha/\gamma)\) which are defined by the equilibrium concentrations at the \((\gamma:\gamma+\alpha)\) - and \((\gamma+\alpha: \alpha)\)-phase boundaries at the diffusion temperature. During the diffusion process a phase transformation \((\gamma\leftrightarrow \alpha)\) occurs and an interphase boundary moves towards the \(\gamma\)-phase, hence a layer of the \(\gamma\)-phase is transformed to a new \(\alpha\)-phase, which may be named \(\alpha\). It is found that measured concentrations of Cr and Ni at the interface correspond to the limiting concentrations \(C(\gamma/\alpha)\) and \(C(\alpha/\gamma)\) of the phase diagram. Figures 1(c) and (d) are a schematic representation of the couple and a micrograph of a couple heated for 100h at 1158K, respectively. Some porosities are seen at the initial junction. In general, they were disappeared after a sufficient long treatment at higher temperatures.
Fig. 1. Migration of the interphase interface between the $\gamma$- and $\alpha$-phases after a diffusion treatment, and concentration profiles in a Fe-Cr-Ni diffusion couple. (a): Concentration profiles of Cr and Ni measured by an EPMA in a diffusion couple (1518K×145hr). (b): A part of phase diagram of the Fe-Cr-Ni system (70wt%Fe). (c): Schematic representation of the diffusion couple. (d): A typical micrograph showing the phase transformation and interface migration.

The interface displacement was found to follow the well-known parabolic law ($D = At^2$), where $D$ is the distance of displacement, $t$ the diffusion time and $A$ a constant. Relations between them at different heating temperatures are shown in Fig. 2.
3.2 Macroscopic Aspects of Diffusion Couples

Interphase boundaries between a parent γ-phase and a transformed α-phase have been found to be essentially planar and no special structural defects have been detected, excepting a so-called "diffusion-induced twins" grown frequently in the γ-phase and in the vicinity of the interface, which are similar to those found in the diffusion couples of α/β-brass and α/β-AgZn bicrystals by one of the present authors and his colleagues/1,2,7/.

Macroscopic structures of the different types of couples, P/P, S/P, P/S and S/S, are schematically shown in Fig.3. They can be summarized as follows:

- P/P, S/P: The grain boundaries of the α₀-phase extended into the αₙ-phase, and reached to the interface.
- P/S, S/S: There appeared no grain boundaries in the αₙ-phase. The crystallographic orientation of the αₙ-phase inherited that of the α₀-phase, except for the special case that will be described in detail in later.

All of the results strongly suggest that the orientations of grains in the transformed αₙ-phase are succeeded by those of α₀-phase in the same manner to the usual grain-growth.

3.3 ORs between the γ- and α-phases

ORs between the γ- and αₙ-phases have been measured in P/P and S/P diffusion couples using the computer-aided X-ray Laue Back-reflection method and also a so-called micro-facet etch-pit technique/10/ when grains are too small to use the former method, in order to check whether some special orientation relationships occur or not.

- P/P: Forty pairs have been measured and their ORs were calculated. It was found that there are no special ORs like the KS or NW relations and no concentrated distributions of ORs, though 7% of ORs was estimated to be near the KS relation within 6 degrees from the ideal KS relation.
- S/P: There appeared no special and preferential ORs, at least, in thirty six pairs examined whose αₙ-phase grains are located in a transformed layer of good contact. However, it is noted that the KS relations deviated within about 4 degrees have been often found only at the edge portions of the bonding.

This fact suggests that a nucleation of αₙ-phase grain may occur at the surface of insufficient contact between the materials. As mentioned in 3.2, in P/S and S/S couples α₀ crystals were grown into the αₙ-phase, i.e. there is very little possibility of the nucleation.
However, when the initial bonding OR between the crystals was set to be a near KS relation, new grains of the an-phase have been nucleated, and it is found that the resulting ORs become closer to the KS relation. A couple of γ- and α-phase crystals whose respective surface normals are (111) and (011) was prepared. Then, they were carefully aligned in a press in order to attain a near KS relation and heated. It was found that both of the grain growth and the nucleation occurred in the transformed phase. Metallographic features in the nucleation zone are shown in Fig.4. Columnar grains nucleated in the an-phase are clearly seen.

ORs between the γ-, α₀- and an-phases were determined. Axis-angle pair describing the OR between the γ- and α₀-phases was calculated to be 42.3°(0.06, 0.16, 0.99) which is deviated from the KS and NW relations by 5.2° and 7.3°, respectively. ORs between the parent γ-phase and several an-phase grains were determined using the micro-facet etch pit technique. Every grain was found to rotate closer to the KS relation by 0.5 degrees. On the other hand, diffusion couples having the deviations more than 9 degrees from the KS relation showed no evidence of such a nucleation. The fact means that deviation of about 5 degrees from the preferential OR is a critical condition for the nucleation.

Fig. 4. A typical transformed phase in a s/s diffusion couple. The crystallographic orientation relation between the γ- and α₀-phases has a small deviation from the K-S relation. (1348K×100hr)

4 - DISCUSSION AND CONCLUSION

The present results indicate that the interphase boundary determined by an arbitrary bonding is generally maintained and migrates during the phase transformation. It can be said that the an-phase is formed by a resultant grain growth of the α₀-phase. However, when the crystallographic orientation between the γ- and α₀-phases is near the preferential OR forming a low energy interface, a situation occurs such that nucleation of an-grains can reduce the interfacial energy in the system. It can be concluded that when \( \Gamma(\gamma/\alpha₀) > \Gamma(\gamma/an) + \Gamma(an/\alpha₀) \) nucleation occurs, but in the opposite case grain growth does, where \( \Gamma(\gamma/\alpha₀), \Gamma(\gamma/an) \) and \( \Gamma(an/\alpha₀) \) are the interfacial energies between the phases defined, respectively.

Another possibility for the occurrence of nucleation observed in the present study may be due to a bad contact between the crystal surfaces where some oxidation or contamination will rapidly take place at the early stage of the diffusion bonding. The frequent occurrence of nucleation observed at the bonding edge may be explained by this phenomenon. While, dezincifications at the high temperatures in the CuZn and AgZn couples investigated/1,2,4,7/ is thought to be a main factor for the nucleation. Some diffusion couples of the α/β-brass have been reexamined supplementally. It has now appeared that most of the non-KS relation is formed by the grain growth of the β-phase. In this case, a density of porosities between the βₙ- and β₀-phases is found to be much lower than that at the interface between a nucleated grain and the old phase. Condition of the contact between the materials is also a significant factor in controlling the transformation mechanisms.

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