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Enhanced Diffusion in Shock Activated Be-Al Interfaces

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Résumé: Une augmentation de la diffusion de l'aluminium dans le beryllium activé par choc a été observée. Des échantillons cylindriques de barreaux de beryllium revêtus d'aluminium ont été soumis de manière axissymétrique à un choc jusqu'à 40 GPa et une déformation résiduelle totale jusqu'à 6.7%. Les défauts dans la microstructure dus à la fois par l'onde de choc et la déformation permet au transport de l'aluminium dans le beryllium de dépasser la saturation de son état solide à l'équilibre. Cette super saturation en aluminium apparaît, après chauffage, à une température relativement faible et forme des interfaces très résistantes.

Abstract - Enhanced diffusion of aluminum in shock activated beryllium has been observed. Cylindrical samples of aluminum coated beryllium rods were axissymmetrically loaded up to 40 GPa and a total residual strain of up to 6.7%. The defect microstructure produced by both the shock wave and strain enabled the transport of aluminum in beryllium to exceed its equilibrium solid state saturation. This "super saturated" aluminum, upon heating, exsolves out at relatively low temperatures and forms very strong interfaces with pressure mated components.

1. INTRODUCTION

The major driving force for this work was to improve/enhance fabrication of beryllium components that are diffusion bonded. The use of diffusion bonding for components that can not be welded or brazed, can be enhanced by shock activation. The obvious advantage of diffusion bonding is its potential for lower thermal treatments in component fabrication. Normal diffusion is time-temperature and microstructure dependent and the ultimate solubility will be characteristic of the system being considered. Therefore, desirable enhancement for diffusion bonding would be to reduce either temperature or time or both for the required process. Additionally, if solubility can significantly be increased, higher strengths of the bond could be expected. Earlier high static pressure work on the Al-Si equilibrium phase diagram [1] has shown that pressures up to 50 Kbar have a dramatic effect on the solubility of Si on Al from ~1 at. % (1 atm.) to 7 at. % (50 Kbar). Also an increase of the eutectic temperature from 577°C (1 atm.) to 677°C (50 Kbar), as well as almost tripling the eutectic point reaction from 12.1 at. % (1 atm.) to 29 at. % (50 Kbar). This is shown in Fig. 1. Based on these results it was decided to pursue similar such effects in Be. Traditionally Al and Al alloys have been used as a "braze" material because of its much lower melting temperature relative to beryllium. Aluminum is but one of only two simple eutectic systems with Be and is shown in Fig. 2 [2], the other one being silicon. Silicon was not considered because of its relatively high eutectic temperature and its brittle nature. Additionally, silicon has a very low solubility in beryllium.

Defects in crystal structures can generally enhance diffusion of second element constituents, particularly if the homologous temperature of the second element is much lower than the matrix which contains the atomic defects. This is an ideal situation for the Al-Be system in that Al has its melting point at 0.60 of that for Be. Thus, if the Be were defected by shock loading, these defects would not appreciably be annealed out at the temperatures used for the diffusion of the aluminum. Consequently, the hope that Al would have enhanced diffusion in microstructurally defected Be.
Fig. 1 Influence of high pressure on the Al-Si composition diagram, after [1].

Fig. 2 The Al-Be equilibrium diagram, after [2].

Fig. 3 Schematic of the shock holder assembly used for the Al-Be experiment.
Thus shock loading concomitant with controlled strains of intimate contact Al/Be interfaces were performed to measure this effect.

2. EXPERIMENTAL METHODS

Beryllium rods having a nominal purity of 99.9% and of 3.0 mm in diameter and 6.7 cm in length were vacuum vapor deposited with Al (nominal purity of 99.99%) on its outer circumference to a thickness of approximately 10 μm. This rod was then inserted into a 1100 Al sleeve having a 9.5 mm outside diameter which in turn was inserted into a 304 stainless steel holder assembly having a 3.8 cm outside diameter and is illustrated in Fig. 3. The holder assembly was then incorporated into a axisymmetric shock assembly as shown in Fig. 4. The actual strain rate, which can be deduced from the hydrocode plots were in the range of 10^6/s and the shock pulse duration’s were calculated to be less than 1 μs and have been described earlier [3-5]. The shock and the associated controlled strains are shown in Fig. 5a, b for two separate samples investigated. The shock pressures were obtained from a 2D eulian hydrocode and the strains were measured using photo printed circle grids on the 1100 Al sleeve. The circle griding technique is described elsewhere [5-8]. Samples were then cut from the post shocked rods at specific positions along its length. Each sample is listed with their associated pressures and strains, these values were obtained as described above and are illustrated in Fig. 5a, b. These parameters are given in table I. In these experiments the shock pressures ranged from 4 to 40 GPa in the Be with a concomitant strain of approximately 0 to 6.7% at the high pressure end for the first sample and 0-3.4% for the other having the same pressure range. After shock loading the samples were extracted and characterized as to the amount of Al transported across the interface and into the Be rod. The amount of Al in Be (and Be in Al) was measured by Energy Dispersive Analysis (EDX) and wavelength spectroscopy. To study the exsolution profiles of the Al, heat treat temperatures used in this investigation on post shocked samples were 645°C (solid state) for all the samples as well as 680°C (melt) on the highest strain portion (most defected) of the Be samples. These were also characterized by EDX and wave length spectroscopy.

3. RESULTS AND DISCUSSIONS

Characterization of the Al and Be were made on the eight samples listed in Table I. Beryllium transport into Al was not detected in any of the shocked samples. However, Al transport was detected to varying concentrations at and near the Be interface and are also listed in Table I. Using the data of Table I, a plot of the effect of strain at essentially constant shock pressure as a function of Al transported into the Be at the interface is shown in Fig. 6. Evident from this plot is the obvious effect of both pressure and strain. Even the effect of low shock pressure and low strain produced more than a 15 fold increase in Al concentration. While the intermediate shock pressure, though somewhat higher strains, essentially doubles the Al concentration over that of the low pressure and low strain. The effect of high shock pressure has a dramatic effect over that of the low and intermediate shock pressure, even at similar strains (sample 8, at 3.4% strain). The Al concentrations increased more than five fold over that of the low pressure and low strain. However, roughly doubling the strain to 6.5%, at 40 GPa, the Al concentration increases to 0.6 wt. % This in essence is about a 100 fold increase over the room temperature equilibrium concentration. One must keep in mind that while this is a phenomenal increase, it only occurs within the first few microns of the outer diameter of the Be rod.

Solid state heat treatment was under taken to observe the diffusion of Al. The concentration gradient of Al across the Al/Be interface into the Be rod is shown in Fig. 7. Evident from this figure is a general increase in Al concentration above the as shocked values when heat treated at 645°C for 1 hr. Not only has the Al increased at the measured distance from the interface, it has diffused further into the Be rod in concentrations well above that for equilibrium, up to a depth of 8 μm. Further past the interface and into the Be (8-16μm) the Al concentration approaches that of the equilibrium saturation at 645°C. A possible explanation for the Al concentration increases after heat treatment can be explained as a result of the increased defected microstructure resulting from a
combination of both the shock pressure and associated strain of the Be. This would enhance lower energy diffusion paths that remain available for the Al for quite some time as the heat treatment temperature is only 0.59 of the melt temperature of the Be. Fig. 8 shows the results of this Al exsolution from samples 4 and 4a. Sample 4 had undergone a 40 GPa shock pressure and a concomitant strain of 6.5%, its Al concentration started out with 0.6 wt. % Al at the interface, and after heat treatment at 645°C for 1 hr., increased to 0.8 wt. % Al. However, at a depth of 10μm from the Al/Be interface the 0.05 wt. % Al concentration when annealed further, continued to decrease, moving closer to that of the equilibrium concentration of ~0.008 wt. %. When the sample was heated to 680°C (Al melt) for 15 min., the 0.05 wt. % Al concentration at a depth of 10 μm decreased to 0.01 - 0.018 wt. % Al. The resultant solubility of the melted sample is much less but still more than equilibrium saturation of Al in Be. The lower solubility is attributed to the natural exsolution of the Al from the Be via the higher driving force (higher temperature) towards equilibrium. Consequently, this high non-equilibrium solubility of Al in Be is indeed the driving force necessary to promote lower temperature-time diffusion bonding.

To test the diffusion enhancement of these shocked and strained samples, a few samples from the rods were machined down then ground and fine sanded down to their Be diameters. They were then reinserted into an 1100 Al disk and then heat treated as before (645°C for 1 hr.).
resulting diffusion bond passed the normal punch test which showed that the interface was intact and had shear failed in the 1100 Al.

TABLE I. Amount of Aluminum in Beryllium before and after heat treatment for shock and strain conditions listed.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Shock Pressure GPa</th>
<th>Strain %</th>
<th>wt. % Al in Be (post shocked)</th>
<th>wt. % Al in Be after 1 hr. at 645°C</th>
<th>wt. % Al in Be after 15 min. at 680°C</th>
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<td>1</td>
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<td>~1</td>
<td>.05</td>
<td>.01</td>
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<tr>
<td>2</td>
<td>34</td>
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<td>.15</td>
<td>.017</td>
<td></td>
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<td>.8</td>
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<tr>
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<td>.6</td>
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<tr>
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</tr>
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<td>40</td>
<td>3.4</td>
<td>.20</td>
<td>.066</td>
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</tbody>
</table>

Fig. 6 Effect of strain as a function of aluminum transport into beryllium at the interface for essentially constant post shock pressures.

Fig. 7 Concentration gradient of aluminum across the Al/Be interface.
4. CONCLUSIONS

The present investigation was concerned with enhancing diffusion of Al in shock activated Be. Shock/strain activation of Be did significantly enhance AI mass transport. Which upon thermal treatment enhances thermal diffusion, thus allowing for the use of lower temperatures or shorter times at higher temperatures. There is a strong correlation between pressure/strain and Al transport into the near surface of the Be in post shocked material. The amount of transported Al was greatest for the highest pressures and strains. The Be/Al post shocked and strained samples, bonded under thermal treatments for all shock conditions tested. While only a few punch tests were carried out on the highest pressure/strained samples the diffusion bonded interfaces were stronger than the 1100 Al.

Fig. 8 Solubility's in the Al/Be system. A-A is the equilibrium saturation of Al in Be.

REFERENCE: