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Effect of atmospheric control during MA-HIP process on mechanical properties of oxide dispersion-strengthened Cu alloy

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In this study, mechanical properties of Dispersion Strengthened (DS)-Cu-Al (aluminum) and Zr (zirconium) alloys, which were fabricated by an MA-HIP method, were investigated for application to the heat sink materials of fusion reactors. The effect of air exposure before the HIP process was studied using a NIFS-Sealing Device. Cu-Al specimen with air exposure before HIP was broken during wire-electrical discharge formation. Cu-Al specimen without air exposure exhibited high fracture strength, but without yielding. Cu-Zr specimen, both with and without the exposure, yielded and exhibits elongation. An increase in yield and tensile strength by approximately 61% and 45%, respectively, were obtained for Cu-Zr specimen by avoiding air exposure. The results showed that Cu-Zr specimen is less susceptible to the atmosphere of the MA-HIP process than Cu-Al.

Keywords: Copper alloy, Mechanical alloying, Dispersion strengthening, Mechanical properties

1. Introduction

Copper (Cu) alloys have been considered as candidates for the heat sink materials for fusion reactors because of their very high thermal conductivity. In ITER, precipitation strengthened (PS)-Cu alloys such as Cu-Cr-Zr are planned to be used [1-4].

However, PS-Cu alloys are well known to be subject to instability of microstructure at high temperature, resulting in significant loss of strength, and irradiation induced softening and hardening. Thus, strengthening of the copper alloys has been required for their application to the divertors of DEMO and nuclear fusion reactors which should possess high heat removal capability at high temperature and radiation resistance [5-7].

On the other hand, dispersion strengthened (DS)-Cu such as Glidcop® (Cu-Al₂O₃) is considered to be an attractive material with respect to higher stability in microstructure and strength at high temperature relative to PS-Cu [8-11]. However, the conventional DS-Cu has insufficient mechanical properties which partly are caused by inhomogeneity and coarsening of the dispersed particles and anisotropic microstructure and properties originated from the fact that the dispersed particles were produced by internal oxidation and extrusion [12].

In our previous our study, we proposed a process combining Mechanical Alloying (MA) and Hot Isostatic Pressing (HIP), which is well known as the process employed for Oxide Dispersion Strengthened Steels (ODSS) [13] in order to improve the properties of Cu alloys [14].

In this study, Cu-1 wt. % Al and 1 wt. % Zr alloys were produced by a process based on the MA-HIP. As a reference study, there was an earlier effort to fabricate

DS-Cu using MA and HIP in which additions of pure-yttrium metal and yttrium oxides were carried out [15].

Our MA-HIP system has unique characteristics which enable the entire the production process from MA to HIP to be carried out under atmospheric control in glove boxes. This makes it possible to investigate the effects of MA-HIP atmosphere on the property of the product. In the present study, we carried out productions and characterizations of Cu-1wt. % Al and Cu-1wt. % Zr alloys, with and without air exposure before HIP.

The purpose of this study is to evaluate the effects of processing atmosphere on the properties of Cu-Al and Cu-Zr alloys produced by the MA-HIP process.

2. Experimental procedure

Cu-1.0 wt. % Al and Zr (labeled as Cu-Al and Zr) alloys were produced by MA and HIP method. The starting materials were powders of pure-Cu, pure-Al, and pure-Zr supplied by Fukuda Metal Foil and Powder Co. LTD. and Yamaishi Metal Co. LTD, CHEMETALL GNBH Co. LTD, respectively. The information of the powders supplied by the vendors is listed in Table 1.

Table 1 Data of raw powders

Material	Purity (%)	Particle size distribution (%)			
		<75 μm	75-106 μm	106-150 μm	150 μm<
Cu	99.8	21.5	36.5	33.5	8.5
Al	99.98	12.9	56.3	30.3	0.5
Zr	98.5	100	0	0	0

The initial elements were mixed and mechanically alloyed in a planetary-type ball mill using 5 mm stainless steel balls in a 250 cc MA pot with ball-powder ratio of 7:3, and the alloying was carried out with a rotating rate of 250 rpm for 32~40 hrs in a pure argon gas atmosphere. The rotation was repeated by an automated program (1hr milling +3hr pause) to avoid excess temperature increase of the pot. The mechanically alloyed powders were then loaded into a mild steel capsule, and were sealed using a NIFS-Sealing Device for HIP shown in Fig.1. The device can weld hose end and round edge of the capsule in a pure argon atmosphere, where the oxygen concentration was maintained below 10 ppm constantly throughout the experiment.

(a) Device appearance
(b) Sealed mild steel capsule
(c) Illustration of NIFS-Sealing Device and encapsulation procedure

The sealed capsules were degassed at 500 °C for 1 hr in 0.1 Pa vacuum. The powders were sintered by HIP at 1223 K for 1 hr with a pressure of 150 MPa. It should be noted that, throughout the process, the materials were not exposed to the air. For comparison, Cu-Al and Cu-Zr powders were processed in the same manner with MA of 32~40 hr in air. After sintering, each specimen was cut to dimensions of 4 mm x16 mm x0.5 or 0.25 mm for tensile tests shown in Fig.2-a. The tensile strengths were assessed at room temperature and with a displacement rate of 1×10^{-3} mm/s using a compression type tensile fixture shown in Fig.2-b and Fig.2-c. The fixture can load tension to the specimen by external compression.

(a) Shape of tensile specimen (mm)
(b) Tensile fixture (fixed condition)
(c) Tensile fixture (tensed condition)

Assessment of the fracture surface was performed by FE-SEM analyses. Metallographic and structural analyses were performed using an optical microscopy (OM) and scanning electron microscopy (SEM).

3. Results

The effect of air exposure before HIP on tensile properties is shown for Cu-Al and Cu-Zr in Fig.3. The data on fracture strength of Cu-Al with air exposure are not available because the specimens were broken during the wire-electrical discharge formation. On the other hand, Cu-Al without air exposure process exhibited high fracture strength but without yielding. The strength achieved 366MPa.

The results on Cu-Zr are also affected by the air exposure before HIP treatment. It is to be noted that the Cu-Zr products have yield strength, and exhibit elongation. The yield and tensile strength of Cu-Zr are 172 MPa and 246MPa with the exposure, and 278 MPa and 358MPa without the exposure, respectively. An increase in yield and tensile strength by about 61% and 45% were obtained, respectively, by avoiding air exposure. The results clearly shows that Cu-Zr is less susceptible to air exposure than Cu-Al.

Fig 4. shows stress-strain curves of Cu-Al and Cu-Zr with and without exposure of air, this curve is one of the typical tests in each set of 10~15 specimens group. Although Cu-Zr exhibit ductile S-S curve, Cu-Al exhibit brittle S-S curve. The elongation of average of 10~15 specimens is shown in Fig 5.

The effect of air exposure before HIP on elongation is shown for Cu-Al and Zr in Fig.5. The data on elongation of Cu-Al with and without air exposure are not available because the specimens did not exhibit plastic deformation. On the other hand, Cu-Zr with and without exposure to atmospheres exhibits plastic deformation. The elongations are 3.4 % and 1.1%, respectively. A reduction of the elongation by about 66% was recognized for the specimens without exposure to air before HIP. However, it should be noted that the result of Cu-Zr with exposure to atmospheres has a large margin of error than that of Cu-Zr without exposure to the atmospheres.

This may be because the micro-structure of Cu-Zr was exposed to atmospheres that were inhomogeneous. As a result, processing using the NIFS-Sealing Device can contribute to the homogenization of a microstructure.

Fig. 6 shows SEM images for cross section and micro-fractured surface of tensile specimens with and without air exposure before HIP after tensile test. In the cases of Cu-Al with air exposure before HIP, the specimens were broken after wire-electrical discharge formation. In the case of Cu-Al without air exposure before HIP, the specimens were successfully formed, allowing tensile tests. The samples did not show reduction of area but exhibited cleavage fracture shown in micro-fractured surfaces.

In the cases of the Cu-Zr with and without air exposure before HIP, the specimens have typical shear lips, which were not shown in Cu-Al in cross-section or in longitudinal section. The micro-fractured surfaces exhibited many dimples which were caused by stretching of shear lips. The results on Cu-Zr show that the alloys are ductile, showing the advantage of Zr addition.

4. Discussion

Significant effects of exposure to air before HIP process were observed for Cu-Al and Cu-Zr in mechanical properties measured by tensile tests at room temperature. The results showed advantages of Zr addition relative to that of Al addition. Here, the difference is discussed with respect to the affinity of oxygen with Al and Zr. A reference study of ODS steel with addition of Al and Zr is available [16], in which it was reported that Zr was preferentially oxidized formation dispersion of Zr oxides. This was justified by the fact that Zr has a higher affinity with oxygen than Al. In the present study, therefore, Zr is thought to have scavenged the excess oxygen more efficiently than Al in Cu matrix.

The results and the discussion suggest that the control of excess oxygen is essential for improving mechanical properties of DS-Cu alloys.

However, the elongation of Cu-Zr is small, and it is necessary to improve the elongation by optimizing the process.

In the future work, we plan to introduce a new process in which oxygen impurity will be reduced by processing in an atmosphere containing hydrogen gas. It is expected that excess oxygen in Cu matrix will be suppressed by this procedure.

5. Conclusions

DS-Cu alloys are promising as heat sink materials of fusion reactors because of the structural stability at high temperature relative to the PS-Cu alloys. Our MA-HIP production system has unique characteristics in which the materials were not exposed to the air in the series of the process. In particular, sealing of the capsules before HIP can be carried out under an atmospheric control in glove boxes called NIFS-Sealing Device. In this study, we investigated the effect of the atmospheric control during the MA-HIP process on the mechanical properties of Cu-Al and Zr alloys. The main results are as follows:

1. Although Cu-Al specimen with air exposure before HIP were broken before the tensile test. Cu-Al specimen without air exposure before HIP exhibited high fracture strength reaching 366MPa. However, no yielding was observed.
2. Cu-Zr specimen exhibited yielding both with and without air exposure. The yield and tensile strength increased by approximately 61% and 45%, respectively, by avoiding the exposure before HIP.
3. Sharp lips and many dimples were observed in cross section analysis of Cu-Zr specimen with and without air exposure before HIP, showing ductile properties, which were not observed for Cu-Al specimen. The results clearly show that Cu-Zr specimen is less susceptible to air exposure than Cu-Al specimen.
4. Although Cu-Al specimen with air exposure before HIP did not exhibit plastic deformation, Cu-Zr specimen exhibited elongation both with and without air exposure. Elongation of Cu-Zr specimen with and without air exposure was 3.4% and 1.1%, respectively. However, the data for Cu-Zr specimen with the exposure had a large margin of error, probably because of the large inhomogeneity of the microstructure.
5. The advantage of Zr addition is expected to be attributed to the fact that Zr has higher affinity with oxygen and can getter excess oxygen in Cu matrix more efficiently than Al. It is essential to control the excess oxygen for improvement of the properties of DS-Cu. A new process is planned to improve the DS-Cu.

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