DISPERSION STRENGTHENED COPPER ALLOYS PRODUCED BY MECHANICAL ALLOYING AND HOT ISOSTATIC PRESSING FOR DIVERTOR APPLICATION

H. Noto *, Y. Hishinuma, B. Huang, T. Muroga

National Institute for Fusion Science, Toki, 509-5292 Japan
*E-mail: noto.hiyoyuki@nifs.ac.jp

Abstract

The realization of advanced fusion reactors rests with improvement of cooling capacity of divertors. Enhancing mechanical properties of Cu alloys is one of the critical issues for the improvement. This paper reports development of dispersion strengthened Cu alloys using ball-milling, encapsulation, and Hot Isostatic Pressing (HIP) facilities. Cu-Al, Zr and Y alloys have been produced so far. The new facilities installed in NIFS made it possible to control oxygen level of the products. In the case of Cu-Y, CuO was added in the middle of the milling for supplying oxygen. These processes resulted in formation of fine microstructures, oxide dispersion, and significant strengthening of Cu alloys.

1. INTRODUCTION

Copper (Cu) alloys are known as attractive heat sink materials of divertors mainly because of their good thermal properties. However, insufficient high temperature strength is the critical issue. Commercial dispersion strengthened (DS)-Cu alloys, which are produced by internal oxidation and extrusion, have higher strength at elevated temperature. These DS-Cu alloys, however, have issues of coarsened dispersed particles and anisotropy in mechanical properties. In this study, a new nano DS-Cu alloys were fabricated by combination of Mechanical Alloying (MA) and Hot Isostatic Pressing (HIP) methods. The MA method can produce finely dispersed nano particles with isotropic microstructure. This alloying-sintering process is well known as a technique enhancing mechanical properties at high temperature applied as the process for producing Oxide Dispersion Strengthened Steel (ODSS). Therefore, we expect that MA-HIP process can offer technical advantages also for DS-Cu alloys. The MA-HIP process is composed of MA, sealing of capsule, and HIP treatment. In general, the sealing of capsules is carried out in air. In this study, we tried to process entirely in controlled atmosphere using a NIFS-Sealing device (NSD), which includes a TIG welding machine in a glove box. The purpose of this study is to investigate the effect of the processing atmosphere on the properties of DS-Cu produced by MA-HIP method.

2. EXPERIMENTAL PROCEDURE

As new nano-DS-Cu alloys, Cu-1.0 wt. % Al and Zr (labeled as Cu-Al and Zr) alloys were produced by MA and HIP method [2]. 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. In this way, Cu-Al and Cu-Zr with different oxygen levels were produced. 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. After sintering, each specimen was cut to dimensions of 4 mm x16 mm x0.5 or 0.25 mm for tensile tests. The tensile strengths were assessed at room temperature and with a displacement rate of $1x10^{-3}$ mm/s using a compression type tensile fixture. Microstructure and microchemistry of the alloys were examined by a scanning transmission electron microscope (STEM) equipped with an energy-dispersive X-ray spectroscope (EDS).

3. RESULTS

3.1. EFFECT OF OXYGEN CONCENTRATION FOR CU-AL AND CU-ZR

Although Cu-Al specimens with low oxygen level were broken before tensile tests, those with high oxygen level exhibited high tensile strength reaching 366 MPa. Cu-Zr specimen exhibited yielding both with low and high oxygen level. The yield and tensile strength increased by about 61 % and 45 %, respectively by the increase in the oxygen level (Figure1). The tensile strength of these alloys are higher than those of annealed and aged Cu-Cr-Zr (~300 MPa), but the elongation was <4 %, lower than that of Cu-Cr-Zr (10 %<) [2]. The measurement of RRR (Residual Resistance Ratio) for Cu-Al with high oxygen level showed that the thermal conductivity of the product is similar to that of pure Cu (Oxygen Free Copper) [1].

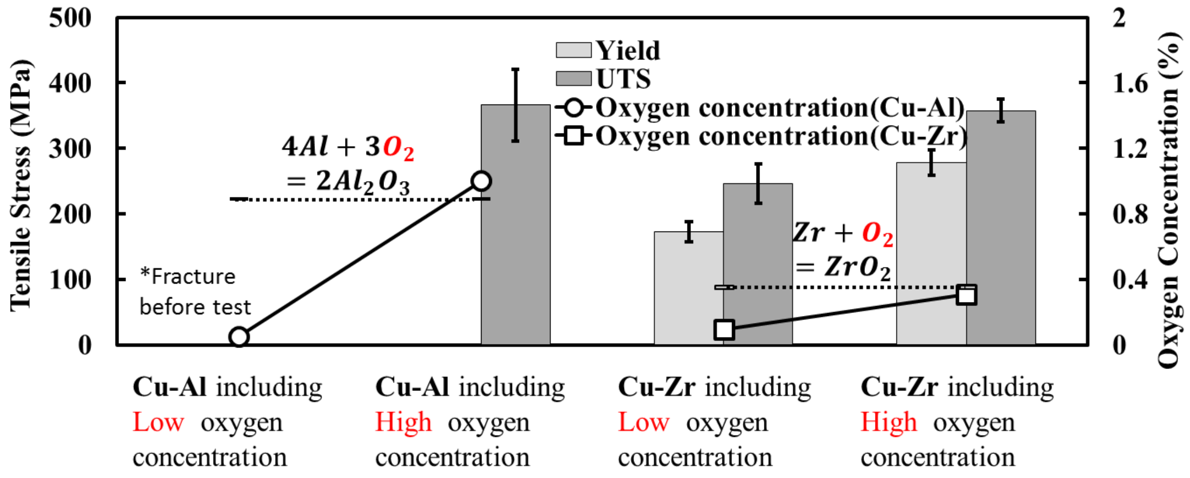

Figure1. Effects of oxygen concentration on mechanical properties and microstructure for Cu-Al and Cu-Zr.

The TEM (Transmission Electron Microscopy) of Cu-Al, Zr specimens confirmed dispersion of Al or Zr oxide compounds in Cu matrix when the oxygen level is high (Figure 2). This means that the Al and Zr oxide particles contributed to strengthening the Cu alloys.

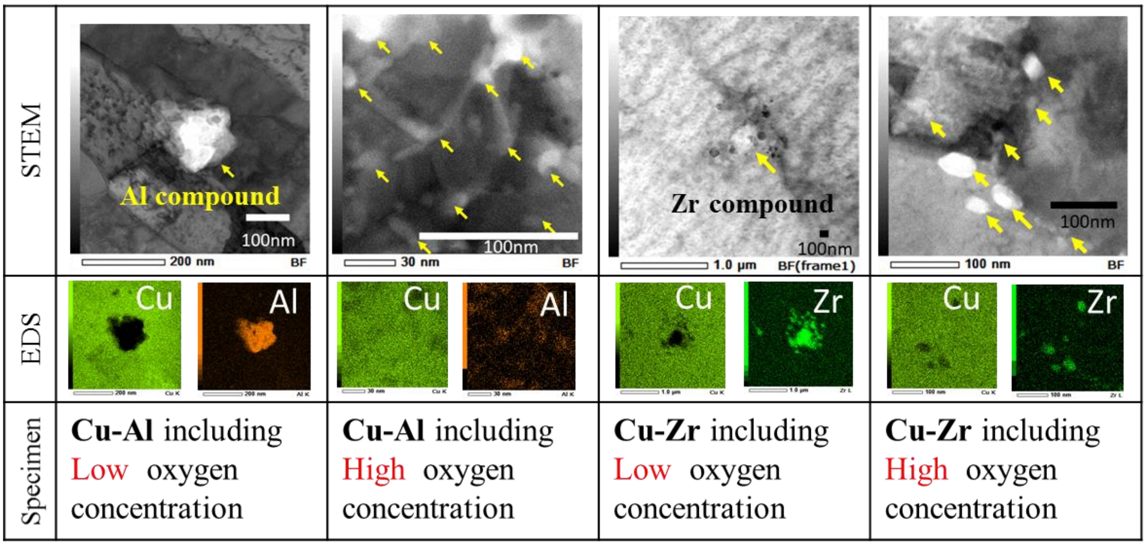

Figure2. STEM image and EDS chemical mapping for Cu and Al, Zr for the alloy with MA for 32~40 hrs and HIP.

3.2. IN-SITU FABRICATION OF CU-Y_2O_3 ALLOY

According to the above results, showing the importance of the oxygen control for production for nano-particles, a new processing was carried out for Cu-1.0 wt. % Y alloy production. In this process, CuO powder were added into Cu-Y alloy powders during MA to adjust the oxygen content. MA and HIP conditions were the same as those for Cu-Al and Cu-Zr. First, Cu and Y powders were mixed and mechanically alloyed for 16 hrs. Second, the mechanically alloyed Cu-Y powders were further milled together with CuO powders for another 16 hrs. During the MA, powders were sampled from the pot periodically and were subjected to X-ray diffraction (XRD) analysis. The results are summarized in Figure 3. It is found that MA of Cu and Y powders for 16 hrs lead to formation of a Cu(Y) solid solution, accompanied by lattice expansion and grain refinement. When the Cu(Y) solid solution powder was milled with CuO powders, reaction between Cu(Y) and CuO occurred, with the lattice shrinking. XRD result of the consolidated sample after HIP verified the successful formation of Y_2O_3 particles. All of the processes can be summarized as follows:

$$3\ CuO + x\ Cu + 2\ Y = (3+x)\ Cu + Y_2O_3 \qquad (1)$$

where x controls the amount of the doping phases. In this report, x was chosen as 112 to produce nominal Cu-3wt%Y_2O_3 alloy (Stoichiometric ratio of raw powders: Cu-2.36wt%Y-3.17wt%CuO).

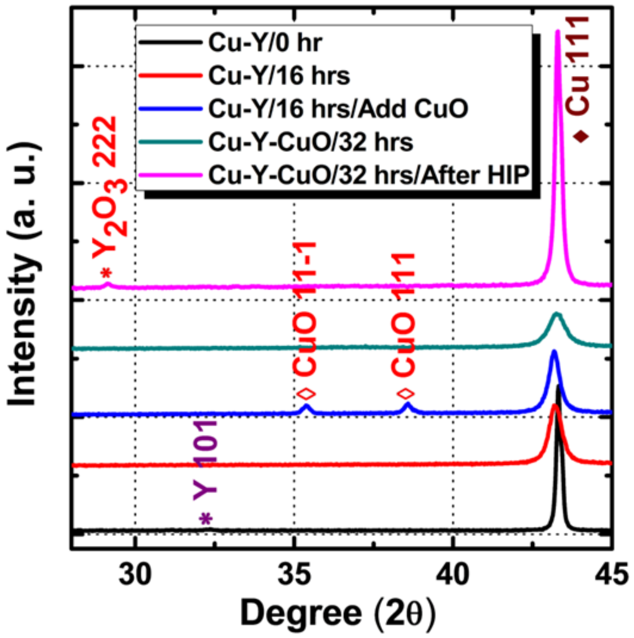

Figure 3. XRD spectra showing the evolution of crystal information of the Cu-Y and Cu-Y-CuO powders during the MA-HIP process.

4. SUMMARY

Fabrication and characterization of new DS-Cu alloys by MA-HIP process were carried out. Fine grain structure and dispersed nano-sized oxide compounds in Cu matrix were observed, enhancing mechanical strength. The study demonstrated that the dispersion strengthening by MA-HIP process can be applied to Cu alloys. The main results are following:

1. Effect of oxygen concentration for Cu-Al and Cu-Zr: Prototype- two DS-Cu (Cu-Al, Zr) adopted dispersion strengthening method was produced. The "oxygen concentration" affected to "mechanical properties" and "increase of dispersed particles. The results means that the particles contributes as a strengthening particle, and effective oxidation of elements (Al, Zr) is important for increase of the particle.

2. In-situ fabrication of Cu-Y_2O_3 alloy: For the effective oxidation of element (Y), new process adding oxidant (CuO) on the process of MA is introduced. The micro-structure including yttrium oxide was obtained.

REFERENCES

[1] T. Yamada, H. Noto, Y. Hishinuma, T. Muroga, H. Nakamura, Development of a dispersion strengthened copper alloy using a MA-HIP method, Nucl. Mater. Energy 9 (2016) 455-458.

[2] H. Noto, T. Yamada, Y. Hishinuma, T. Muroga, Effect of atmospheric control during MA-HIP process on mechanical properties of oxide dispersion-strengthened Cu alloy, Fusion Eng. Des. 124(2017)1024-1027.