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Microstructure and mechanical properties of mechanically alloyed ODS copper alloy for fusion material application

S.M.S. Aghamiri[a,*], N. Oono[a], S. Ukai[a], R. Kasada[b], H. Noto[c], Y. Hishinuma[c], T. Muroga[c]

[a] Graduate School of Engineering, Hokkaido University, Sapporo 060-8628, Japan
[b] Institute of Advanced Energy, Kyoto University, Kyoto 611-0011, Japan
[c] National Institute of Fusion Science, Gifu 509-5202, Japan

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ABSTRACT

Advanced oxide dispersion strengthened copper alloys are promising structural materials for application in divertor system of future fusion reactors due to high irradiation resistance, high thermal conductivity, and good mechanical properties. In this study, a new ODS copper including 0.42wt%Y_2O_3 nanosized oxide particles was developed successfully by mechanical alloying method using addition of 1 wt% Stearic acid in Ar atmosphere. Mechanical alloying resulted in decrease of crystallite size to 28 nm in concurrent with increment of dislocation density and hardness to the saturated level of 1.7 $\times 10^{15} m^{-2}$ and 226$HV_{0.1}$ after 48 h milling, respectively. Consolidated ODS copper by SPS and then hot roll-annealing at 900 °C/60 min showed an average grain size of 1.1 μm with a near random texture. Furthermore, TEM observations demonstrated fine semicoherent Y_2O_3 oxide particles distributed with a misfit parameter (δ) of 0.17 in copper matrix with an average size of 10.8 nm and interparticle spacing of 152 nm. Finally, tensile test evaluation determined comparable mechanical properties of the annealed ODS copper (Cu-0.42wt%Y_2O_3) with Glidcop-Al25 including a yield strength of 272 MPa and total elongation of 12%, by two mechanisms of grain boundary strengthening and oxide particle strengthening.

1. Introduction

Advanced copper alloys are the main candidate for high heat flux and high temperature materials application in different components of fusion energy reactors such as divertor due to extremely high energy density. Key properties of high thermal conductivity (~300–400 W/m.K), good mechanical property (yield strength >200 MPa) and high irradiation resistance (especially irradiated-condition ductility) are essential for effective performance of these materials in a high heat flux, high irradiation environment [1].

Up to now, two main category of copper alloy have been developed for fusion application: precipitation strengthened (PS) copper alloys and dispersion strengthened (DS) copper alloys [1–4]. PS copper alloys such as CuCrZr (used in ITER reactor) or CuNiBe can be strengthened considerably by fine precipitation of second phase particles. The main problem of PS copper alloys is softening by coarsening of precipitates at intermediate to high temperatures. DS copper alloys have the benefit of keeping the strength even at elevated temperatures by dispersion of fine stable oxide particles. The main commercially available dispersion-strengthened copper alloys are Glidcop-Al15, Al25, Al60 and MAGT 0.2, all produced by internal oxidation method. These alloys are strengthened by dispersion of fine alumina particles inside the copper matrix during their manufacturing process. However, both developed copper alloys have the serious limitation of low ductility after irradiation [5,6].

Oxide dispersion strengthened (ODS) alloys are the attractive option for the irradiation environments because of good mechanical properties and irradiation resistance. It has been proved that dispersion of fine Y_2O_3 oxide particles in ODS Steels have an important role to keep the mechanical properties and good irradiation resistance by capturing of defects and reducing irradiation swelling [7,8]. Up to now, the best developed way for good dispersion of Y_2O_3-based oxide particles in different ODS materials is mechanical alloying (MA) [8,9]. However, there is only few studies in literature regarding mechanical alloyed (MAed) ODS copper alloys with a limited progress [10–13]. It is expected to promote the mechanical properties by appropriate dispersion of fine oxide particles in a well-designed copper matrix. To develop new ODS copper with superior properties by MA method, at first, it needs to overcome the severe sticking of milled powder induced by high ductility of copper. Cryo-ball milling by liquid nitrogen has been reported to be used as successful milling method for dispersion of nanosized yttria and calcia in copper alloy [14,15]. In this study, a room temperature

* Corresponding author.
E-mail address: sms.aghamiri@gmail.com (S.M.S. Aghamiri).

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milling was studied in an alloy with a nominal composition of Cu-0.5wt % Y_2O_3 by addition of processing control agent (1 wt% stearic acid (SA)) during milling in Ar atmosphere. After consolidation followed by hot rolling and subsequent annealing at 900 °C, the mechanical properties of the ODS copper alloy were evaluated based on microstructural characterization.

2. Experimental

In this research, a high purity (99.9%) copper powder (particle size of ~50 μm) and Y_2O_3 nanopowder (particle size of ~50 nm) were used as raw materials. The powder mixture was mechanically alloyed by using Fritch-P6 planetary ball mill with different additions of stearic acid up to 2 wt% as process control agent (PCA) in Ar atmosphere. 1 wt % stearic acid was chosen as the optimum amount based on the higher powder output and purity of MA powder. The ball milling was performed with ball to powder ratio of 10:1 and rotation speed of 470 rpm in different milling times up to 96 h. After the milling, the chemical composition of the powder was analyzed by inductively coupled plasma (ICP) method.

X-ray diffraction was conducted by means of $Cu - K\alpha$ radiation with a Philips X´Pert PRO to characterize the phases during milling. The change in crystallite size (D) during MA can be calculated from XRD broadening based on the Hall–Williamson equation [16]:

$$\beta_s \cos\theta = 2\,\varepsilon\, sin\theta + (K\lambda/D) \tag{1}$$

where β_s is the peak full width at the half maximum intensity (FWHM) (after subtracting the instrumental broadening), θ is the diffraction angle, ε is the local strain, λ is the x-ray wavelength of the Cu-Kα (0.154 nm) and K is the Scherrer constant (0.9), respectively. Moreover, the dislocation density (ρ) can be correlated in terms of local strain (ε) in the following equation [16]:

$$\rho = 7.14\left(\frac{\varepsilon}{b}\right)^2 \tag{2}$$

where b is Burger's vector (2.52×10^{-10} m). By measuring θ and β_s for the planes of (111), (200), (220), (311) and (222), the crystallite size (D) and dislocation density (ρ) were calculated and plotted versus milling time. This plot was accompanied with the average Vickers microhardness data of the milled powders measured by a HMV-Micro Hardness Tester-SHIMADZU, under a load of 980 mN by a diamond pyramid indenter and dwell time of 30 s with 10 times repetition. Vickers microhardness measurements were performed on each milled powder in different milling times after mounting the powder into the epoxy resin and polishing the surface.

The MAed ODS copper powder after 48 h milling was consolidated by spark plasma sintering (SPS) at 900 °C/45 min. Then the material was hot rolled at 900 °C with a 50% thickness reduction and a reduction rate of 0.5 mm per pass followed by annealing at 900 °C/60 min. The powder and bulk samples of ODS copper were observed by FESEM (JEOL JSM-6500F) equipped by electron back scatter diffraction (EBSD) detector. To attain inverse pole figure (IPF) and Orientation distribution function (ODF), the EBSD data were analyzed by OIM software. The Sample preparation for EBSD was done by grinding with SiC paper up to 2000 grit and then polishing with 1 μm diamond paste and final polishing with silica suspension. A JEOL JEM-2010 transmission electron microscopy was used to study the oxide particles in the microstructure in high magnification with a voltage of 200 kV. Furthermore, a FEI-Titan STEM (300 kV) equipped with high-angle annular dark field (HAADF) detector and EDS analysis was used to analyze elemental distribution of fine oxide particles. The thin samples for TEM observation were prepared by focused ion beam (FIB) (JEOL JIB-4600F). For evaluation of oxide particle distribution, the interparticle spacing (λ) was derived from the below equations based on TEM images [9]:

$$\lambda = 1.25 l_s - 2r_s \tag{3}$$

$$l_s = \sqrt{\frac{2\pi\overline{r^3}}{3f\overline{r}}} \tag{4}$$

$$r_s = \frac{\pi\overline{r^2}}{4\overline{r}} \tag{5}$$

where, r is the particle radius obtained from the TEM images. $\overline{r}$ is the average oxide particle radius, and $\overline{r^2}$ and $\overline{r^3}$ are the averages of r^2 and r^3, respectively. The volume fraction of the dispersed oxide particles was estimated to be 0.0075 and the average distance between the centers of oxide particles is l_s. The average particle size is considered as D ($2r_s$).

In order to evaluate mechanical properties, tensile test was performed at room temperature under a strain rate of 1.0×10^{-10}/S using Shimadzu, SSL-1KN tensile machine. Miniaturized size specimens for the tensile test were prepared with a gauge dimension of 5 mm in length, 1.2 mm in width and 0.5 mm in thickness by using an electro-discharge processing machine.

3. Results and discussions

3.1. Microstructure of ODS copper powder processed by MA in Ar atmosphere

Fig. 1 shows the x-ray analyses of Cu-0.5wt%Y_2O_3 powder between 0 and 96 h milling times in Ar atmosphere. In these curves, in addition to peaks of copper matrix, the peaks of carbon compounds related to released PCA (Stearic acid) and yttrium oxide can be observed (as shown in the insets by arrows). However, yttrium oxide peak is hard to distinguish after 24 h milling due to probable decomposition during high energy milling as discussed in different ODS materials [8]. Another point in this figure is broadening of copper peaks with increasing milling time which is the characteristic behavior of high strain mechanically milled powders caused by repeated cold welding, fracturing and rewelding of powder particles during milling [17].

Fig. 2(a) shows the evolution of crystallite size and dislocation density during increasing milling time calculated by Eqs. (1) and (2). The curve demonstrates that crystallite size of powder particles decreases sharply in a short time and reach to the stable size of ~28 nm after about 48 h. In other hand, trend of dislocation density shows a concurrent increase with decreasing crystallite size during milling time and saturates in the level of $1.7 \times 10^{15} m^{-2}$ after 48 h. Change of Vickers microhardness of milled powders at different milling times in Fig. 2(b) determined consistent result with Fig. 1, i.e. increasing the hardness and reaching to $226HV_{0.1}$ after steady state condition of 48 h milling.

Fig. 3 illustrates the ODS copper powder after mechanical alloying for 48 h in Ar atmosphere. As shown in this figure, a fine and loose powder with irregular-shaped particles of ~100 μm average size was obtained after the high energy milling. The milling conditions resulted in a high powder recovery of 94% without agglomeration and sticking of ductile copper powder to milling cup by using optimum amount of 1 wt% stearic acid which balanced the rate of cold welding and fracturing of powder particles [17]. The chemical composition of the mechanical alloyed ODS copper powder has been specified in Table 1. This composition indicates a copper alloy containing 0.42 wt% Y_2O_3 oxide particles which suggests dissipation of some oxide powder during evacuation of MA pot atmosphere to replace with Ar. In addition, some minor impurity of iron, Ex. O and carbon were inserted from milling medium, atmosphere and releasing of PCA during high energy milling, respectively. The solute iron can decrease the thermal conductivity and segregation of carbon and Ex. O has been reported to result in intermediate temperature embrittlement [1,13]. So, to reach a sample with higher purity and hence higher thermal conductivity, some modifications in MA and thermal treatment in hydrogen environment [17] need to be done.