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Characterization of γ-U–10 wt.%Mo alloy powders obtained by hydride-milling-dehydride process

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ABSTRACT

Uranium–molybdenum alloy powders are raw material of uranium alloy fuels prepared by powder metallurgic process in nuclear reactor. U–10 wt.%Mo alloy powders were prepared directly by hydride-milling-dehydride process without γ phase to α phase transformation under long time heat treatment, the powders were studied by XRD and SEM. During absorb–desorb hydrogen cycle, the absorbed hydrogen quantity of U–10 wt.%Mo alloy increased with the cycle number increasing, after eight cycles, the absorbed hydrogen quantity reached a stable value, and absorbed hydrogen was saturated in alloy. U–10 wt.%Mo alloy hydride was milled in argon atmosphere, then, alloy hydride were heated to desorb the absorbed hydrogen at 600 °C, after these processes, U–10 wt.%Mo alloy powders were obtained. XRD results show that U–10 wt.%Mo alloy powders was still γ phase, its indicated that during preparation, the alloy was always body centered cubic structure, phase transformation process did not exist. SEM observation showed that the particle shape of U–10 wt.%Mo alloy powders was nonuniform, but particle size was less than 50 μm, some were below 10 μm.

1. Introduction

It is well-known that U–Mo dispersion fuel in γ-phase state is considered as a perspective fuel in frames of Reduced Enrichment for Research and Test Reactors (RERTR) program due to its high irradiation stability and corrosion resistance [1–6]. At present time the set of new high density uranium alloys is under investigation, among which there are U–Mo alloys with different Mo content. To manufacture dispersion fuel elements on the basis of these alloys, it is necessary to develop effective powder production methods. Among the number of existing powder production methods, there are several most perspective ones which can be suitable for high density uranium alloys, such as mechanical crushing (milling, grinding, etc.) [7–10], atomization [11–13], as well as hydride–dehydride (HD) process [7,14–16]. Among these powder production methods, HD process is widely used, because U–Mo powders obtained by HD process have less impurities, moreover, different powder particle sizes can be attained by raw material particle size and hydride–dehydride cycle number controlling. But before HD process, in order to quicken U–Mo alloy hydrogen-adsorption reaction rate, U–Mo alloy must be heat-treated to make α phase separated out at grain boundary, after hydrogen-desorption, it must be heat-treated again to revert to γ phase. The HD process become complex because of the alloy heat treatment, further more, heat treatment needs much time, and powder production period was prolonged.

In this study, U–10 wt.%Mo alloy powders were directly prepared by hydride-milling-dehydride (HMD) process without γ phase to α phase transformation under long time heat treatment, and the powders were characterized by XRD and SEM for application as a dispersant for research reactor fuel elements.

2. Material and methods

U–10 wt.%Mo alloy are prepared by vacuum induction melting. The alloy samples, provided by Zhi-gang Wang et al., are pieces cutted from alloy ingot. Before HMD process, oxidation layer on alloy surface was removed in nitric acid until the surface presented metallic luster, then, the acid was remove in de-ionized water and anhydrous alcohol, after alloy surface was dried by an argon gas flow, 155 g U–Mo pieces were put in hydrogenation reactor, all the operations were carried out in glove box full of argon.

Before hydrogen-adsorption, the reactor was pumped out to less than 40 Pa at room temperature to remove gas impurities adsorbed on reactor wall, then, it was pumped out to less than 4 Pa at 250 °C and kept 30 min to remove the absorbed water, finally, it was pumped out to less than 1 Pa at 600 °C and kept 1 h to make fresh alloy surface revealed and quicken U–10 wt.%Mo alloy hydrogen-adsorption reaction.

The hydride–dehydride cycles of U–10 wt.%Mo alloy were carried out in an experimental system shown in Fig. 1, the

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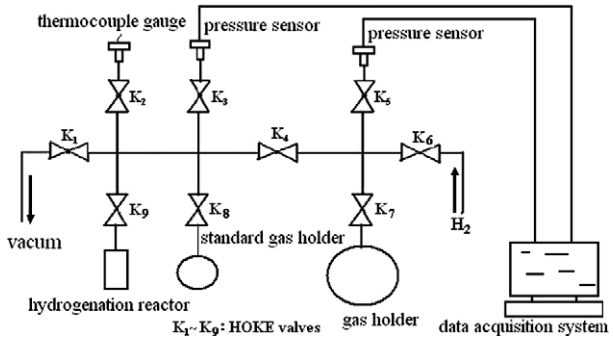

Fig. 1. Schematic diagram of experimental system.

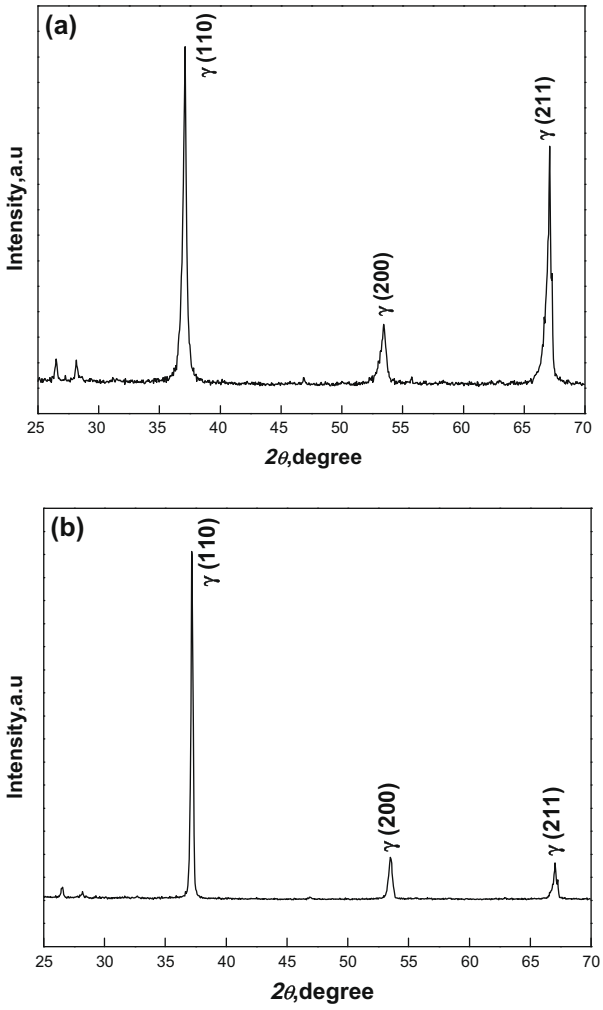

Fig. 2. XRD pattern of U–10 wt.%Mo alloy: (a) vacuum induction melting ingot and (b) after polished.

hydrogenation reactor was stainless steel with one end closed by flange. Hydrogen-adsorption reaction were carried out at room temperature, initial pressure was 0.5 MPa, the quantity of adsorbed-hydrogen was measured by a standard gas holder and pressure sensor. After the adsorbed-hydrogen quantity was saturated, U–10 wt.%Mo alloy hydride was heated at 600 °C to desorb hydrogen, and hydride–dehydride process was repeated. When the adsorbed-hydrogen quantity reached a stable value, U–10 wt.%Mo alloy hydride was milled in argon atmosphere, then, the hydride

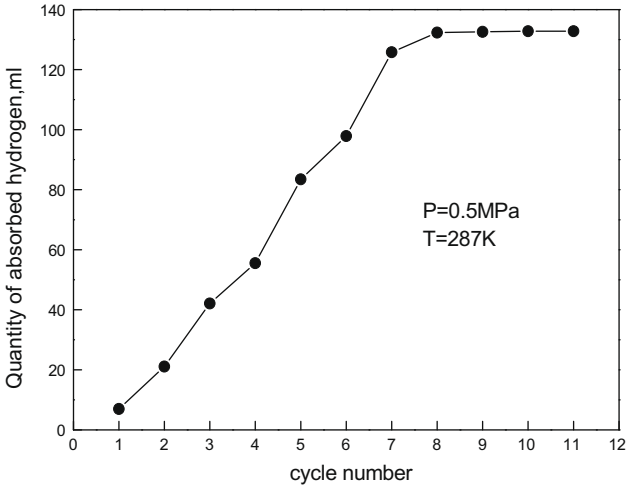

Fig. 3. Relationship between absorbed hydrogen quantity of U–10 wt.%Mo alloy and cycle number.

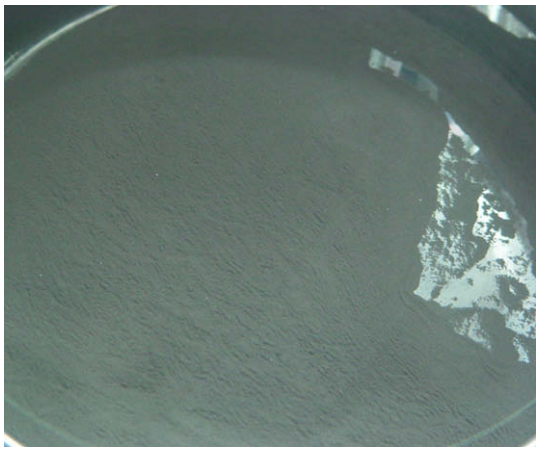

Fig. 4. Photograph of U–10 wt.%Mo alloy powder.

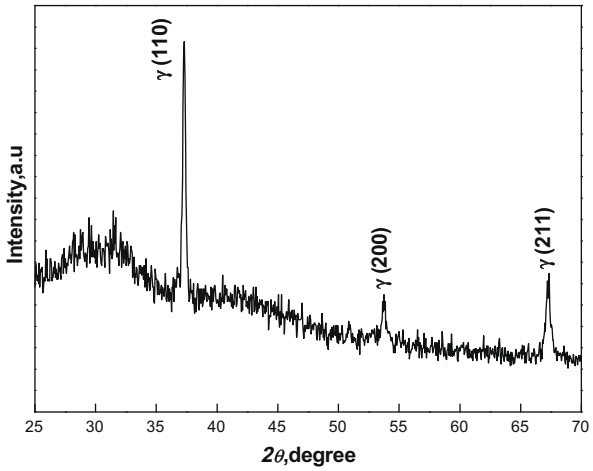

Fig. 5. XRD pattern of U–10 wt.%Mo alloy powder.

was heated at 600 °C to desorb hydrogen, and the U–10 wt.%Mo powder was obtained.

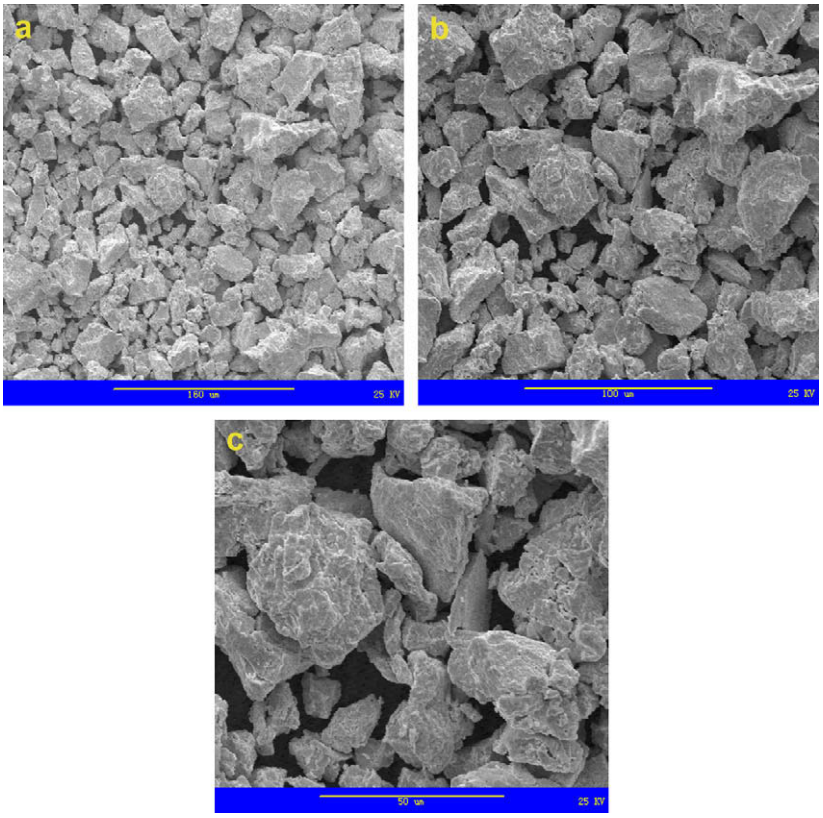

Fig. 6. SEM micrographs of U–10 wt.%Mo powder: (a) ×300, (b) ×500 and (c) ×1000.

U–10 wt.%Mo alloy ingot of vacuum induction melted and the powder were analyzed by X-ray diffraction, using the Cu Kα wave length. The morphology and microstructure of the powder particle size were characterized with a scanning electron microscope (SEM).

3. Results and discussion

In case of U–10 wt.%Mo alloy ingot prepared by vacuum induction melting (as shown in Fig. 2a) and after polished by No. 600 carborundum paper (as shown in Fig. 2b), the XRD pattern revealed the complete body centered cubic γ-uranium formation.

In Fig. 2a, there were two diffraction peaks when diffraction angle was between 25° and 30°, it was due to the oxidation layer on the alloy ingot surface, after the ingot surface was polished by carborundum paper, the oxidation layer was removed, and these two peaks disappeared, as shown in Fig. 2b.

γ phase U–10 wt.%Mo alloy sample was put in hydrogenation reactor, the hydride–dehydride cycles were carried out. The relationship between absorbed hydrogen quantity of U–10 wt.%Mo alloy and cycle number was shown in Fig. 3. It was observed that the absorbed hydrogen quantity of U–10 wt.%Mo alloy increased with the cycle number increasing, finally, it reached a stable value. The sample surface was treated before hydride, but there was still quite thin oxidation layer, therefore, the absorbed hydrogen quantity was little at the beginning. After hydrogen-adsorption, the alloy swelled and desquamated from the matrix alloy, then, fresh alloy surface revealed and the absorbed hydrogen quantity increased. Eventually, absorbed hydrogen was saturated in alloy and the absorbed hydrogen quantity reached a stable value.

After the absorbed hydrogen quantity of alloy reached a stable value, the U–10 wt.%Mo hydride was put in a stainless steel milling can in glove box full of argon, and milled for 5 min in argon atmosphere, then, the hydrogen in milled alloy was desorbed in hydrogenation reactor at 600 °C. The U–10 wt.%Mo powder obtained as shown in Fig. 4.

The XRD pattern of U–10 wt.%Mo powder (as shown in Fig. 5) indicate that it was still body centered cubic γ-uranium formation after HMD process.

The shape of the U–10 wt.%Mo powder as observed by scanning electron microscope (SEM) is shown in Fig. 6, the shape of all the powder particles and the particle sizes were nonuniform. But all the particle size was below 50 μm, some were below 10 μm.

4. Conclusion

In order to develop nuclear fuel with γ phase U alloy, U–10 wt.%Mo alloy powders are produced by a hydride-milling-dehydride method. The characteristics of the HMD process and the alloy powders investigated are as follows:

1. U–10 wt.%Mo alloy powders were obtained by HMD process, γ phase to α phase transformation under long time heat treatment was not needed, the HMD process is simple and feasible.
2. At the beginning, absorbed hydrogen quantity of U–10 wt.%Mo alloy is quite little, but it increases with the hydride–dehydride cycle number increasing.
3. XRD analysis of U–10 wt.%Mo alloy ingot and powders indicated that γ phase to α phase transformation under long time heat treatment did not exist during HMD process.
4. SEM observation of U–10 wt.%Mo alloy powders revealed that all the particle size was below 50 μm, some were below 10 μm, but the shape of the powder particles and the particle sizes were nonuniform.

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