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Preparation and characterization of sintered Mo-Re alloys

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Abstract

By the method of powder metallurgy, we have tried to fabricate Mo-Re alloys, which were electron beam weldable. Severe quality control was carried out during the whole fabrication process focused to reducing oxygen contamination. It is inevitable that the starting raw powders of Mo and Re were both high purity with 99.99 mass% up. Moreover, high vacuum sintering was performed before final sintering with high-purity hydrogen gas. As a result, we obtained electron beam weldable Mo-Re alloys, the total oxygen content of which was about 10 mass ppm or less, respectively. Several specimens were melted by electron beam welding (EBW) method. It was found that EBW gives an easy and effective survey to examine the weldability and the quality of the materials. Fracture surfaces examined by AES exhibited very low content of oxygen, carbon and nitrogen or that less than detectability limit. In conclusion, we have succeeded to obtain defect-free welds of sintered Mo-Re alloys. Furthermore it was found that Mo-Re alloys showed excellent potentialities not only in mechanical properties at low temperature but also in the respects of microstructure.

1. Introduction

Due to the beneficial effect of Re on the ductility of refractory metals, Mo-Re alloys are well known as one of the candidate materials for high temperature application such as diverter or protective parts in CTR, components in contact with liquid metals in FBR and nuclear reactor in future aerospace plane project. Although high purity Re powder is itself very expensive, sintered Mo-Re alloys are more attractive than melted ones not only from a standpoint of cost-performance but also in the respect of potentiality of high performance. In the previous report(1), a relatively large amount of oxygen was found to be the main cause for a poor weldability of electron beam method in the sintered Mo-Re alloys containing 2, 4, 10, 13 and 40 mass% Re. In this study, therefore, a new fabrication process was developed to reduce oxygen contamination of the sintered Mo-Re alloys containing 15, 20, 25, 30 and 50 mass% Re. Microstructure and mechanical properties were characterized by metallography, chemical analysis, hardness, bend test, SEM/EDX, XRD, AES and EBW.

2. Experiment

Fig.1 illustrates the flow chart of the fabrication process. High Purity

molybdenum and rhenium powders of 99.99 mass% up were used to produce the pressed and sintered billets. A rubber bag mold was filled with the blended powder mixture and isostatically pressed to form a small bar. The green bar was sintered in flowing hydrogen with a dew point(d.p.) less than 233K at 1273K for 7.2ks, followed sintering in vacuum under 3×10^{-3} Pa at 1773K for 36ks. Final sintering was performed in flowing hydrogen with a dew point less than 213K at 2113k for 144ks. The sintered ingot was rolled to the sheet of about 1 mm thickness using a conventional molybdenum rolling machine. Hot and warm rolling were employed to reduce the gauge for finish rolling at ambient temperatures. Chemical composition of the sheets was determined by the following methods. Na, K and Re were analyzed by Flame Absorption Spectroscopy. Carbon, oxygen and nitrogen were analyzed by combustion method. Other elements were analyzed by Inductively Coupled Plasma Emission Spectroscopy (ICP-ES). Several specimens were melted by electron beam welding(EBW) method to examine the weldability and the quality of the materials. One hour annealing properties of the as-rolled sheets were examined by optical metallography, scanning electron microscopy combined with energy dispersive analyzer (SEM/EDX) and hardness test. Mechanical properties were compared by the bend test. The microstructures and the fracture surfaces of sintered Mo-Re alloys were characterized by optical metallography, X-ray diffraction(XRD), SEM/EDX and Auger electron spectroscopy(AES).

Fig.1 Flow chart of the fabrication process for electron beam weldable sintered Mo-Re alloys.

3. Results and discussion

The chemical analysis of the starting powders provided by the manufacturer and the as-rolled Mo-Re alloys is shown in Table 1. Mo-15Re, Mo-20Re, Mo-25Re and Mo-30Re prepared by a new fabrication process contain 15.9%Re, 21.4%Re, 25.6%Re, 31.7%Re, respectively. Chemical compositions of other elements were as follows; Al, Si, Fe, Sn, Pb ≤ 10 ppm, Cr, Ni, Cu < 5 ppm, Na, K, Mg, Ca, Mn ≤ 1 ppm, C = 10 ppm, N = 1-2 ppm, O = 7-11

ppm. On the other hand, Mo-2Re, Mo-4Re, Mo-10Re and Mo-13Re contain 1.7%Re, 3.3%Re, 8.8%Re, 12.0%Re, respectively. Other elements were Al $\leq$ 11 ppm, 30 < Si < 60 ppm, 30 < Fe < 50, Sn, Pb, Cu and Mn < 3 ppm, 8 < Cr < 9, 10 < Ni < 13, Na and K = 1 ppm, 4 < Ca < 7, 2 < Mg < 3, 6 < C < 13 ppm, 1 < N < 9 ppm. O = 30, 49, 25, 19 ppm, respectively. Mo-40Re contains 37.0%Re. C, N and O were not analyzed in the as-rolled condition. But C and O was 12 ppm and 66 ppm, respectively, in the specimen annealed at 1673K for 3.6ks. In a previous study(1), oxygen was remarkably increased among other elements, which largely corresponded to an increase of Al and Fe. It suggests that such a contamination was occurred during sintering and rolling. Main features of a new fabrication process are summarized as follows; [1] Starting raw powders of Mo and Re were both high purity with 99.99 mass% up. [2] High vacuum sintering was performed before final hydrogen gas sintering. [3] High purity hydrogen gas was used during the sintering. [4] Severe quality control was carried out during the whole fabrication process focused to reducing oxygen contamination. Mo-50Re alloy including a relatively higher Re content showed σ phase in the matrix examined by XRD. Impurity level was similar to that of Mo-Re alloys containing 15 to 30 mass% Re. As a result, we obtained Mo-Re alloys containing 15 to 50 mass% Re, the total oxygen content of which was about 10 mass ppm or less, respectively. Several specimens were melted by electron beam welding method to examine the weldability and the quality of the materials(2)(3). Corresponding to the chemical composition, especially to the oxygen content, welding defects such as blow holes and porosities were observed in the materials containing high total oxygen.

Table 1 Chemical composition(mass ppm) of the starting powders and the sintered Mo-Re alloys.

	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Pb	Si	Sn	Na	K	C	N	O
Re		1		<1	9	<1				3		<3	<5			
Mo	4	2	8	3	30	7	<3	11	<3	<15	<3	1	8			
15Re	<10	<1	<5	<5	10	<1	<1	<5	<10	<10	<10	<1	<1	10	2	11
20Re	<10	<1	<5	<5	10	<1	<1	<5	<10	<10	<10	<1	<1	10	1	10
25Re	<10	<1	<5	<5	10	<1	<1	<5	<10	<10	<10	<1	<1	10	1	7
30Re	<10	<1	<5	<5	10	<1	<1	<5	<10	<10	<10	<1	<1	10	1	8
50Re	<10	<1	<5	<5	10	<1	<1	<5	<10	<10	<10	<1	<1	<10	6	12
0Re	<3	<2	<8	<3	20	<0.8	<3	5	<3	<15	<3	1	1	5	6	18
2Re	11	5	9	<3	50	2	<3	11	<3	30	<3	1	1	10	3	30
4Re	7	4	8	<3	30	2	<3	10	<3	30	<3	1	1	13	9	49
10Re	9	7	9	<3	50	3	<3	13	<3	30	<3	1	1	8	2	25
13Re	11	7	9	<3	60	3	<3	11	<3	60	<3	1	1	6	1	19
40Re	83	14	10	<3	150	2	<3	20	<3	40	<3	1	1	12	1	66

Fig.2 shows a typical example of the fracture surfaces of the welds at 77 K. Mo-13Re exhibited significant porosities in the intergranular surfaces, while Mo-20Re showed smooth intergranular surfaces without porosities. It is found that electron welding is a effective indication of the quality of the sintered molybdenum alloys. Fracture surfaces examined by AES exhibited very low content of oxygen, carbon and nitrogen or that less than detectability limit, indicating a good agreement with above result(2). Yield stress in bend test and bend angle indicate strength and

ductility, respectively. Mechanical properties of the sintered Mo-Re alloys were not inferior to those of melted ones. Hardness increased with an increase of Re content. Recrystallized temperature ranged from 1473K to 1873K with an increase of Re content, showing good agreement with that of melted ones(2). In conclusion, a new fabrication process was developed for EB weldable sintered Mo-Re alloys. It was found that Mo-Re alloys showed excellent potentialities not only in mechanical properties at low temperature but also in the respects of microstructure(2)(3).

Fig.2 Fracture surfaces by the bend test at 77K in the as-welded specimens of (a) Mo-13Re and (b) Mo-20Re.

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