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Vacuum hot-pressed beryllium and TiC dispersion strengthened tungsten alloy developments for ITER and future fusion reactors

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ABSTRACT

Beryllium and tungsten have been selected as the plasma facing materials of the ITER first wall (FW) and divertor chamber, respectively. China, as a participant in ITER, will share the manufacturing tasks of ITER first-wall mockups with the European Union and Russia. Therefore ITER-grade beryllium has been developed in China and a kind of vacuum hot-pressed (VHP) beryllium, CN-G01, was characterized for both physical, and thermo-mechanical properties and high heat flux performance, which indicated an equivalent performance to U.S. grade S-65C beryllium, a reference grade beryllium of ITER. Consequently CN-G01 beryllium has been accepted as the armor material of ITER-FW blankets. In addition, a modification of tungsten by TiC dispersion strengthening was investigated and a W–TiC alloy with TiC content of 0.1 wt.% has been developed. Both surface hardness and recrystallization measurements indicate its recrystallization temperature approximately at 1773 K. Deuterium retention and thermal desorption behaviors of pure tungsten and the TiC alloy were also measured by deuterium ion irradiation of 1.7 keV energy to the fluence of 0.5–5 $\times$ 10^{18} D/cm^2; a main desorption peak at around 573 K was found and no significant difference was observed between pure tungsten and the tungsten alloy. Further characterization of the tungsten alloy is in progress.

1. Introduction

Vacuum hot-pressed (VHP) beryllium was selected as the armor material- of ITER first wall (FW) blankets and S-65C beryllium from Materion Brush Beryllium & Composites (USA) was considered as the reference material- in the 2001 final design, owing to its low beryllium oxide content and high elongation rate, as well as good thermal load resistance capabilities [1]. Because China and Russia will share the manufacture of ITER FW blankets, they want to develop their own beryllium to be used in their ITER-FW procurement packages. Up to now the qualification processes for new beryllium materials are complete and Chinese VHP beryllium CN-G01 and Russian beryllium TGP-56FW (1% BeO) were formally accepted as the ITER-FW armor materials in December 2010. The qualification procedure includes two aspects: one is the fundamental physical and thermo-mechanical properties; the other is the thermal responses to the ITER-FW load conditions. The critical acceptance criteria are the equivalence to S-65C beryllium.

On the other hand, high purity tungsten and high quality carbon fiber composite were selected as the plasma facing materials in the ITER divertor chamber in the initial operation phase of ITER. An all-tungsten divertor could be used for D–D and D–T operation phases [2], however, high purity tungsten as the plasma facing material-in the future fusion reactors will face many problems, for example high ductile–brittle transition temperature (DBTT) and related low recrystallization temperature, which will result in machining difficulty and wall load limitation. One possible modification method is to raise the recrystallization temperature by TiC particle dispersion and lower the DBTT by severe plastic deformation [3,4].

In this paper, firstly the fundamental thermo-mechanical properties and high heat load performances of CN-G01 beryllium are discussed, and then the development of a TiC dispersion strengthened W alloy is introduced by focusing on the DBTT and recrystallization temperature measurements, as well as deuterium retention and thermal release behaviors.

2. Experimental

2.1. Fabrication of vacuum hot-pressed beryllium CN-G01 and its main properties

CN-G01 beryllium has been developed since 2005 and its main manufacturing processes are as follows: beryllium pebbles with purity of 99% are fabricated from beryllium mineral, and then cast into beryllium ingots in a vacuum induction furnace. The

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ingot surface is cut off and the remaining ingot is mechanically chipped into small pieces, which are then crushed into beryllium powders by high speed gas steam. After classifying and chemical cleaning, beryllium powders for vacuum hot press are obtained. A VHP beryllium block is sintered in a vacuum hot-pressing furnace. Finally beryllium tiles are produced by cutting VHP beryllium blocks along the pressing direction.

After several years' efforts, the BeO content in CN-G01 was controlled to less than 1% and the total elongation rate at room temperature was more than 3%. Other physical and thermo-mechanical properties were also measured and characterized according to the specification for ITER-grade beryllium; they satisfied the requirements of ITER grade beryllium and were very close to those of S-65C.

2.2. Qualification tests of high heat loads

The high heat flux properties under ITER FW load conditions were characterized by two tests. One is thermal shock response to transient loads by simulating the ITER plasma disruption and edge localization modes (ELMs); in this test single shots and multiple shots at energy densities of 1–5 MJ/m^2 (load area 5 × 5 mm^2, pulse duration 5 ms) were performed by using beryllium coupons with size of 15 × 15 × 5 mm^3. The other is comprehensive tests using Be/Cu mockups (42 × 106 × 30 mm^3 with three Be tiles of 40 × 24 × 10 mm^3) with active cooling to simulate plasma disruption, vertical displacement events (VDE) and thermal fatigue. All experiments were conducted by comparing with S-65C and candidate Russian beryllium.

2.3. Preparation of TiC dispersion strengthened tungsten alloy

The traditional powder sintering method was adopted for tungsten alloy manufacture. First the fine carbon and TiH_2 powder with particle size of about 1 μm were blended by jet milling with tungsten powder of 99% purity and several μm in particle size, and the C/Ti ratio of atomic number is slightly more than 1 owing to the more ablation of C during the subsequent sintering processes. Then the W–C–TiH_2 mixed powder was shaped by cold isostatic pressing and then sintered at a final temperature of 2573 K in an H_2 atmosphere. This sintering process included three key stages. The first stage was from room temperature to 1623 K in which oxygen may dispelled by the action of C and O, then kept at this temperature for half an hour, in which tungsten carbides were preferentially formed. After that, the furnace temperature was rose to 1973 K and kept at this temperature for about 3 h; in this process TiC was formed by the abstraction reaction of the C atoms from the tungsten carbides. Tungsten plates were prepared by hot rolling or forging. The density of the sintering embryo and the control of the in situ chemical reaction of $C + W = W_2C$ (or WC) + TiH_2 = W + TiC + H_2 are the key factors to guarantee the formation of the TiC dispersion phase and successfully hot rolling or forging. At this point, a low-TiC-content W alloy (0.1% TiC) has been prepared and the fabrication technique has been optimized for higher-TiC-content alloy.

3. Results and discussions

3.1. Characterization of CN-G01 beryllium

A qualification program for new beryllium grades was established to assess the possible performance of the new grades. This program included two main aspects:

(1) Fundamental physical and mechanical properties.
(2) Comparative thermal performance tests with respect to the reference grade S-65C.

The program for thermal performance behaviors included several tests such as thermal shock resistance capabilities, vertical displacement event (VDE) simulation testing and subsequent thermal shock tests, and thermal cyclic fatigue tests after VDE simulation testing.

Table 1 lists the data for physical and thermo-mechanical properties of CN-G01 beryllium measured at ambient temperature, in which the data are the average values measured by at least three samples, the measure procedures and the data at elevated temperature can be found in elsewhere [5]. All data are very close to values for S-65C.

Fig. 1 shows the cracking behaviors of CN-G01 and S-65C beryllium under single and multiple thermal shocks; no differences were found between CN-G01 and S-65C. Generally, the cracking and melting initiated at the transient load of about 2 MJ/m^2 for a single shot. When the transient energy load was larger than 2.4 MJ/m^2, the surface cracking and melting became significant as shown in Fig. 1a; one can see that the current through the beryllium coupons fluctuated and even waned owing to the shielding effect of vapor from melting. Furthermore, in the case of multiple shots with the objective to simulate the ELMs, the crack initiated at lower energy density and became serious with increasing numbers of shots (Fig. 1b).

A heat flux with energy density of 40 MJ/m^2 was used to assess the crack behavior under ITER VDE loads. The result indicated that almost all cracks stopped in the melt layer of CN-G01 beryllium, which is similar to that of S-65C beryllium. After VDE load three tiles of CN-G01 beryllium received thermal fatigue tests under 2 MW/m^2 heat flux and 15 s heating time for 1000 cycles; however no obvious difference was found [6]. It also suggested that the thermal stress that originated from 2 MW/m^2 heat load could not supply enough driving energy for crack extension; in fact, qualification tests of Be/Cu/stainless steel joining technology have identified no damage to beryllium and Be–Cu joining by 1.75 MW/m^2 heat flux for 1000 cycles and subsequent 2.25 MW/m^2 for 100 cycles [7]. The present result is certainly desirable since the 1.75 MW/m^2 wall load corresponds to the plasma mode of multifaceted asymmetric radiation from the edge (MARFE) in ITER.

For VHP beryllium, the key factors for the thermal response to high heat loads are the BeO content and the elongation rate. Furthermore, surface treatments could influence its thermal responses, in particular the cracking behaviors. In fact, some deep cracks of about 1 mm were found on the surface of partial beryllium tiles polished by mechanical machining after they experienced 1000 repetitive pulse loads (power density 80 MW/m^2, pulse duration 25 ms and load area 10 × 10 mm^2) proposed by Watson [8] with the aim of fast screening of different beryllium grades by focusing on the crack sensitivity; they were considered to probably be associated with the machining injuries on the beryllium surface since the additional "Watson"-like test identified only about 30 μm deep cracks for all tested beryllium coupons treated by careful mechanical polishing [9]. Before heat load tests, a machine damage layer with twin grain structure and a thickness of approximately 30 μm was observed as shown in Fig. 2; therefore the small cracks are obviously due to the twin grain layer that resulted from mechanical machining. It also suggested that chemical etching may be the best method for surface finishing.

3.2. Characterization of W–0.1%TiC alloy

For the W–TiC dispersion strengthened alloy, the critical parameters are its recrystallization temperature and DBTT. The DBTT was measured by impact tests and the preliminary results indicated