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Swelling, mechanical properties and microstructure of beryllium irradiated at 200 °C up to extremely high neutron doses

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Abstract

The paper reports on an investigation of four beryllium grades (TE-56, TE-30, TIP, DIP) produced in Russia using the technology of hot extrusion (HE) and hot isostatic pressing (HIP). Specimens were irradiated in the SM reactor at a temperature of 200 °C to neutron doses in the range of $(0.7–13.1) \times 10^{22}$ cm^{-2} ($E > 0.1$ MeV) and then subjected to post-irradiation examination. Beryllium swelling, microhardness and mechanical tensile and compression tests results as a function of neutron dose are presented. In addition, the paper contains the results of irradiated beryllium microstructure examination. Swelling increases with dose and does not exceed 4%. Microhardness also increases steadily with neutron dose. The irradiation induced embrittlement of all the tested specimens with increasing dose leads to significant decrease of brittle strength. The void size and density tend to increase in the microstructure of irradiated beryllium.

1. Introduction

At present beryllium is considered as a possible neutron multiplier for the fusion reactor DEMO blanket, where it will be exposed to extremely neutron doses. Irradiation of beryllium in the research high flux nuclear reactor SM under conditions simulating operating conditions of the fusion reactor units permits a preliminary assessment of radiation damage in beryllium. The latest results of beryllium damage examination after low temperature irradiation [1–3] testify to significant degradation of its physical–mechanical properties and serious microstructure damage associated with accumulation of large amount of transmutant helium in beryllium.

The paper presents the examination results of the influence of neutron irradiation on swelling, mechanical properties and microstructure of Russian beryllium grades irradiated in the SM reactor at 200 °C to a wide range of neutron doses. Data obtained lately for highest doses are of special interest as such information is very limited in open literature available data. The operating temperature of beryllium neutron multiplier exceeds the irradiation temperature of 200 °C considered in the paper. But assessment of the state of beryllium irradiated up to extremely high neutron doses with significant accumulation of radiation defects and transmutant

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helium leads to more understanding of radiation damage mechanisms in beryllium.

2. Experimental

Four grades of beryllium produced in Russia using the technology of hot extrusion (HE) and hot isostatic pressing (HIP) were used for the experiment. The chemical composition and grain size of the examined beryllium grades are given in [2]. For mechanical tensile tests specimens in the form of cylindrical dumbbells with 3 mm diameter × 10 mm gauge length were used, and the specimens used for compression tests had the 6 mm diameter × 8 mm gauge length. Specimens of the TE-56 and TE-30 anisotropic grades were cut out from cylindrical blanks in two orientations – along and across the extrusion axis.

The specimens were irradiated in sealed capsules in a helium atmosphere. The capsules were loaded into research channels of the SM reactor core. According to calculations, the temperature of the specimens reached 200 °C. Neutron dose varied in the range of $(0.7–13.1) \times 10^{22}$ cm^{-2} ($E > 0.1$ MeV), corresponding to a damage dose of 4–74 dpa. The calculated maximum helium accumulation in beryllium reached 21 800 appm.

Dimensions of only the cylindrical specimens were measured for swelling assessment. The measurement error of swelling did not exceed 0.1%. Swelling also was calculated by comparing density of the initial and irradiated specimens. Density was measured using the hydrostatic method in the medium of CCl_4. The measurement error in this case was 0.2%. Mechanical tests were performed at temperature equal to irradiation temperature using a displacement of 1 mm/min. Vickers microhardness was measured at room temperature using a 100 g load. The beryllium microstructure was investigated by means of the optical microscope MIM-15 D.

3. Experimental results

3.1. Swelling

Fig. 1 shows swelling of four beryllium grades as a function of neutron dose, measured by change in dimensions and density of the irradiated specimens. All experimental results are plotted in the same diagram since no significant data scattering for different beryllium grades was observed. There is also no essential difference between the results of swelling determination by the specimen dimensions or density. Swelling increases steadily as the neutron dose increases and at the maximum dose of 13.1×10^{22} cm^{-2} ($E > 0.1$ MeV) swelling remains in the range of 2.2–4.3%.

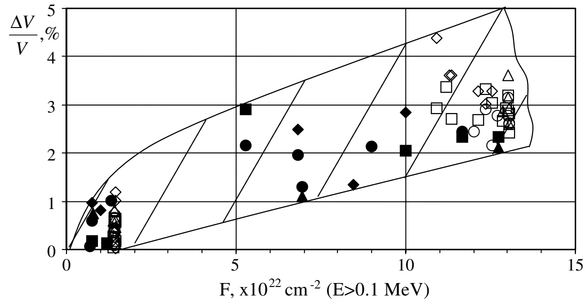

Fig. 1. Beryllium swelling $\Delta V/V$ as a function of neutron dose F: ○ – TE-56, geometrical dimensions; ● – TE-56, density; □ – TE-30, geometrical dimensions; ■ – TE-30, density; △ – TIP, geometrical dimensions; ▲ – TIP, density; ◇ – DIP, geometrical dimensions; ◆ – DIP, density.

3.2. Mechanical properties

Microhardness was determined using a diamond Vickers indentor. The results of not less than 10–15 dimples of the examined specimens are used. Microhardness of four beryllium grades as a function of dose is given in Fig. 2. The increase of microhardness seems to saturate as dose increases and no significant difference in microhardness of beryllium grades is observed. At the highest neutron doses beryllium microhardness is in the range of 6500–8600 MPa, i.e. it exceeds the initial level by several times.

Mechanical tensile and compression tests resulted in brittle failure of all irradiated beryllium

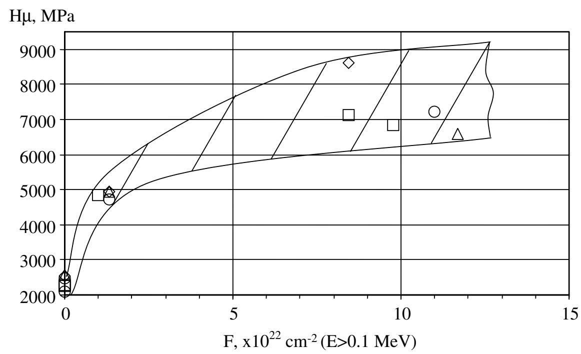

Fig. 2. Beryllium microhardness $H\mu$ as a function of neutron dose F: ○ – TE-56; □ – TE-30; △ – TIP; ◇ – DIP.

specimens. That is why damage-induced stress, i.e. brittle strength, can be a characteristics of radiation damage level of beryllium. The yield strength was determined in the initial state. Fig. 3(a) shows brittle strength of four beryllium grades as a function of dose determined by tensile tests performed at temperatures from 20 °C to 200 °C. It should be noted that brittle strength of the specimens irradiated up to the same neutron fluence at temperatures ranging from 20 °C to 200 °C did not differ greatly. Increase in neutron dose leads to significant decrease of irradiated beryllium brittle strength. There is no marked difference in the effect between the investigated grades, including anisotropic specimens irrespective of whether they were cut out along or across the extrusion axis. At the highest dose the brittle strength remains at the level of 20–100 MPa and no further degradation is observed. Brittle strength of irradiated beryllium tested in compression as a function of dose is similar (Fig. 3(b)), i.e. it decreases abruptly in the dose range of (0.8–1.2) $\times 10^{22}$ cm^{-2} ($E > 0.1$ MeV) and the change saturates at high doses. At a dose of 13.1×10^{22} cm^{-2} ($E > 0.1$ MeV) the beryllium brittle strength varies in the range of 100–800 MPa.

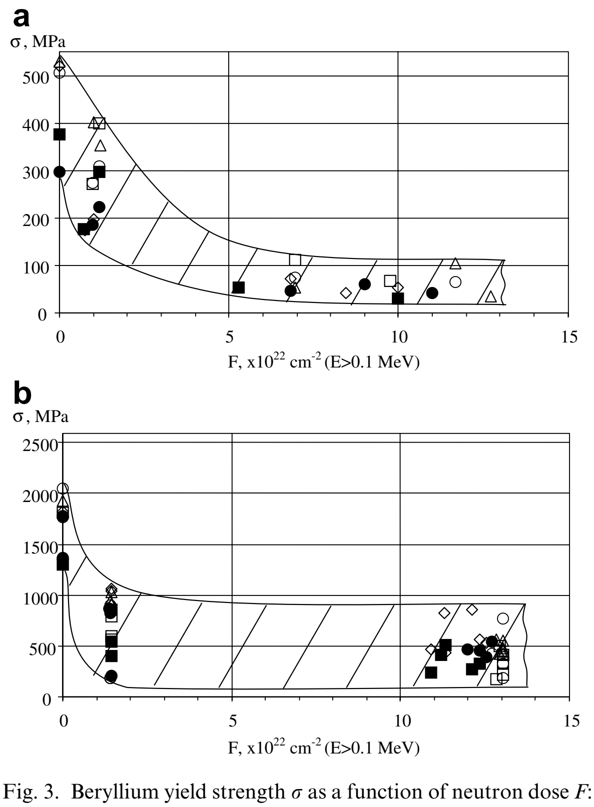

Fig. 3. Beryllium yield strength σ as a function of neutron dose F: (a) tensile tests; (b) compression tests (○ – TE-56, along the axis, ● – TE-56, across the axis, □ – TE-30, along the axis, ■ – TE-30, across the axis, △ – TIP, ◇ – DIP).

3.3. Microstructure

The beryllium microstructure in the initial state is characterized by the presence of voids resulting from hot pressing of beryllium powder, the technology used for the material production. Fig. 4(a) and (b) demonstrates the initial microstructure of the TE-56 beryllium grade fabricated using the hot extrusion technology. The character of the void distribution depends on orientation of the investigated specimen with respect to the extrusion axis. If the specimen is cut perpendicular to the axis, then the voids are distributed quite uniformly along the grain boundaries (Fig. 4(a)). When the specimen is cut parallel to the axis, then the voids basically line up in chains along the grain boundaries stretching along the axis (Fig. 4(b)). The size of the initial voids is 1–2 μm.

Neutron irradiation of beryllium leads to an increase of the void size and density. It follows from Fig. 4(c) and (d) that the microstructure of the TE-56 beryllium grade evolves greatly during irradiation. The void diameter increased up to 15–20 μm, and a net of voids appeared on the boundaries [4]. The character of void distribution for the specimens cut along and across the extrusion axis remained the same. A similar distribution of voids was also observed in the TE-30 beryllium grade, fabricated using the HE technology (Fig. 4(e) and (f)). Beryllium of the TIP and DIP grades, fabricated by the HIP technology, do not demonstrate distinct texture and quite a different void distribution is observed (Fig. 4(g) and (h)). In this case there are no extensive chains of voids in any of the directions. The voids mainly distribute along the grain boundaries, and the character of distribution does not depend on the section plane, selected for examination. The voids of all beryllium grades have the shape of a polygon with smoothed angles.

4. Discussion

Analysis of the swelling and mechanical properties of beryllium irradiated at 200 °C shows (Figs. 1–3) that its properties are prone to serious degradation. It expresses itself as a change of the volume up to 4%, significant (by several times) increase of microhardness, strong embrittlement and, consequently, abrupt decrease of brittle strength. No

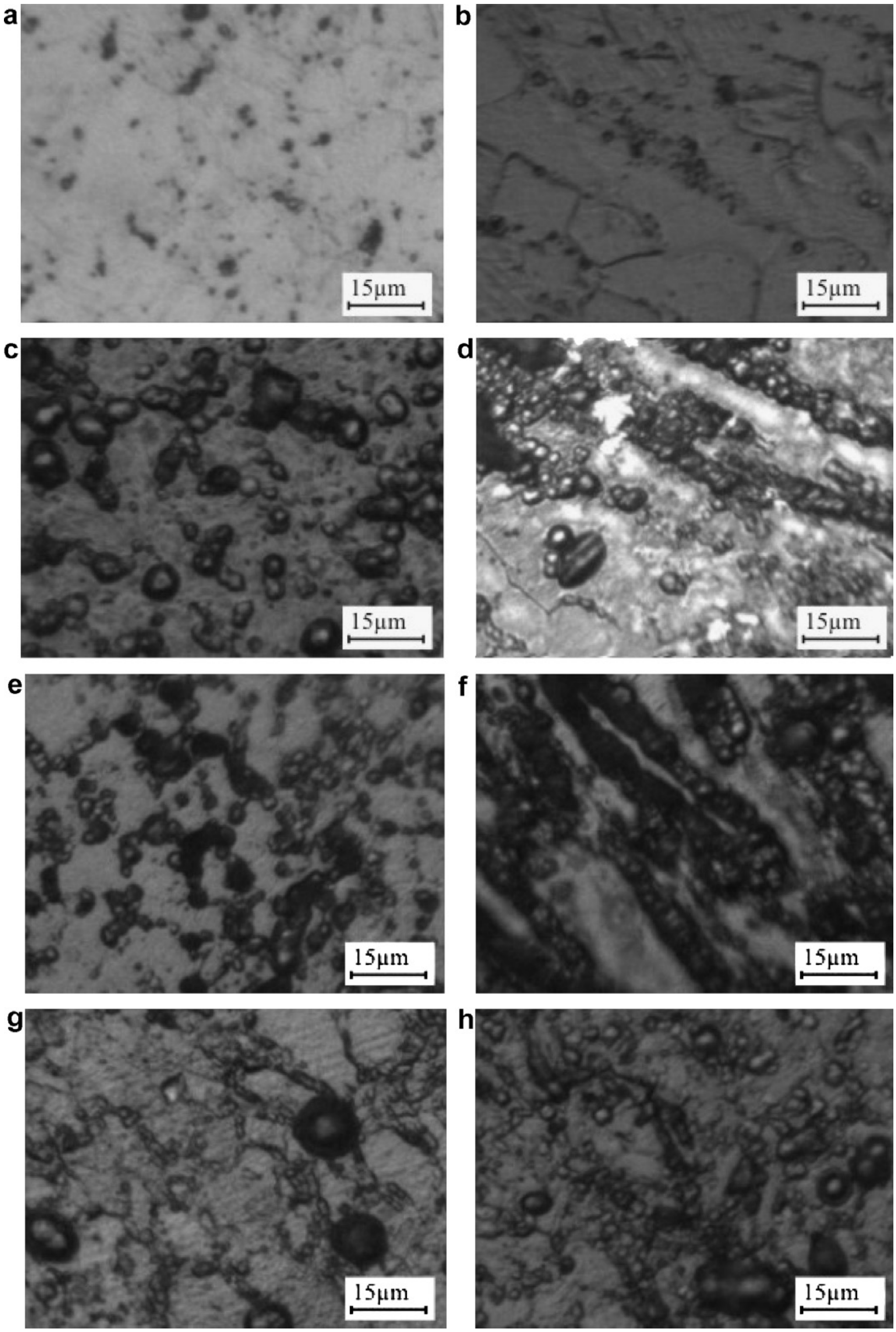

Fig. 4. Microstructure of beryllium: (a) TE-56, across the axis, unirr.; (b) TE-56, along the axis, unirr.; (c) TE-56, across the axis, irr., $F = 9.8 \times 10^{22}$ cm^{-2} ($E > 0.1$ MeV); (d) TE-56, along the axis, irr., $F = 11.0 \times 10^{22}$ cm^{-2} ($E > 0.1$ MeV); (e) TE-30, across the axis, irr., $F = 9.8 \times 10^{22}$ cm^{-2} ($E > 0.1$ MeV); (f) TE-30, along the axis, irr., $F = 8.45 \times 10^{22}$ cm^{-2} ($E > 0.1$ MeV); (g) TIP, irr., $F = 11.7 \times 10^{22}$ cm^{-2} ($E > 0.1$ MeV); (h) DIP, irr., $F = 8.5 \times 10^{22}$ cm^{-2} ($E > 0.1$ MeV).

significant difference was revealed in the state of beryllium fabricated using different technologies and characterized by different initial properties. This is most evident at maximum neutron doses of (10–13) $\times 10^{22}$ cm^{-2} ($E > 0.1$ MeV) when properties of different beryllium grades become similar.

The final beryllium swelling that results from transmutant helium accumulation is determined by the sum of swelling values from separate grains. However, the contribution of each separate grain to the total swelling is not additive. Crystal anisotropy typical of beryllium with the hexagonal close packed lattice leads to anisotropic swelling of separate grains. As a result, when the dose increases and the strength of the separate grains increases, a tendency toward grain bond weakening is observed. The increasing difference between the strength of the grain and the grain boundary results in a total decrease of beryllium strength under external loading. Significant differences apparently lead to a minimization of the differences between different beryllium grades as far as radiation resistance is concerned.

Compact beryllium fabricated from powder by means of hot pressing or other technologies has the same kind of porosity, i.e. cavities along the grain boundaries. The anisotropy of swelling between separate grains leads to an increase of the void size and number due to grain-boundary stresses, resulting from the tendency of the grains to shift relating to one another. Considerable accumulation of helium in beryllium cannot be considered a decisive factor for void generation, since the diffusive mobility of gas atoms under low temperature irradiation is not sufficient for them to migrate a distance comparable to the grain size [5,6]. It is well known, that helium bubbles are generated in low temperature irradiated beryllium only after post-irradiation annealing at a temperature of 500 °C and higher [7]. However, the voids must contain some helium that has reached the boundary from near- boundary areas.

5. Conclusions

1. Several Russian-origin grades of beryllium fabricated using the technologies of hot extrusion and hot isostatic pressing were irradiated in the SM reactor at a temperature of 200 °C up to neutron doses of (0.7–13.1) $\times 10^{22}$ cm^{-2} ($E > 0.1$ MeV) and their swelling and mechanical characteristics were studied. The experiments showed significant degradation of beryllium properties, namely swelling up to 4.3%, microhardness increase up to 6500–8600 MPa, deterioration of strength up to 20–100 MPa after tensile tests and up to 100–800 MPa after compression tests.
2. There is no significant difference between the investigated beryllium grades as far as radiation resistance is concerned. In particular, the tendency of radiation damage saturation is observed at the highest neutron doses.
3. Under irradiation conditions, the grain-boundary voids increase in size and density and a net of void appears. It leads to weakening of the grain boundary and, consequently, to embrittlement and abrupt degradation of the material mechanical strength.

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