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Study of beryllium hardening obtained by powder metallurgy

In the first part of the article the results of the study of powder hardening processes occurring during beryllium powders consolidation by the hot pressing method are shown. The dependences of the content and morphology of the hardening phase depending on the content of low-melting impurities on the sintered Beryllium powder grains surface have been studied. A hypothesis is proposed that explains the transition of an oxide film from an amorphous to a crystalline state – devitrification, and the effect of low-melting impurities on the mechanism of the devitrification process and, as a consequence, on the effect of "dispersion-grain boundary" hardening. This hypothesis is based on theoretical confirmation with the provision of graphic material demonstrating the process of devitrification, accompanied by a dispersed-grain-boundary hardening mechanism. The final results of statistical processing carried out on industrial batches showing the dependence of the impurities content influence on the properties of hot-pressed beryllium are presented. In the second part of the article the results of studying the effect of hardening of beryllium obtained in the process of sintering by the method of hot isostatic pressing (HIP) are shown depending on the temperature of powders consolidation. Based on the results of electron microscopic studies, the dynamics of the reinforcing phase formation at the grain boundaries of sintered beryllium is shown. The quantitative dependence of the precision elastic limit and the conditional yield stress of gas-statically compressed beryllium on the size of the strengthening beryllium oxide particles and the consolidation temperature of the powders have been established. The resulting equation gives a description of the "dispersed-grain-boundary" hardening mechanism of isostatically pressed beryllium. All dependencies are also represented by graphic material reflecting the essence of the research.

Keywords: beryllium, powder, hot isostatic pressing, microstructure, beryllium oxide, pressing temperature, electron microscopy, mechanical properties, dispersion, tensile strength, yield stress.

Introduction

The beryllium industry in Kazakhstan manufactures products mainly using powder metallurgy methods: obtaining powders and further consolidating them into a compact sintered material by hot vacuum and isostatic pressing.

At present beryllium production in Kazakhstan produces sintered beryllium of TGP-56 grade, which, in addition to its chemical composition, is certified for physical and mechanical properties (density, tensile strength, yield strength, elongation). However, the use of beryllium in high-tech industries, such as space, aviation and nuclear technology prescribes a certain level of special properties, in particular, dimensional stability and precision tensile strength. The use of beryllium in gyro-instrument making and metal optics, due to the high specific characteristics of rigidity inherent in it by nature, makes high demands on the dimensional stability of products to be achieved by beryllium production engineers.

One of the characteristics of dimensional stability is the precision tensile strength ($\sigma_{0.005}$), which is about 50 MPa for the best GIP-56 grade of beryllium for instrumental purposes. As research showed [1, 2], the specified characteristic is correlated with the conditional yield stress $\sigma_{0.2}$, which in turn is determined by

the grain size in accordance with the Hall-Petch dependence. Since the grain size of compact sintered beryllium obtained by powder metallurgy methods is determined first of all by the particle size of the initial powder, the main way to increase these characteristics is to increase the dispersion of the initial powders and control the content of beryllium oxide in them. However, an increase of the cost price, pyrophoricity of powders, dustiness of industrial premises, the content of impurities, deterioration of technological properties, etc. was observed in this case and limited the possibilities of production and the use of highly dispersed Beryllium powders with a dispersion of less than 30 microns.

It is also known that the strength properties of beryllium including the precision tensile strength increase with an increase in the content of strengthening phases along the grain boundaries of beryllium oxide first of all. Beryllium oxide is formed as a film several nanometers thick on the surface of beryllium particles in the result of oxidation during mechanical grinding of ingots. As shown in papers [3–7] the mechanism of formation of reinforcing beryllium oxide particles consists of two stages: cracking of the beryllium oxide film when the powder is heated to 700–750°C (due to the difference in the values of the thermal expansion coefficients of the oxide and metal) and coagulation of oxide particles upon further heating and compaction of the powders. It was also shown that the presence of low-melting impurities such as Al, Si, Mg in beryllium affects the process of beryllium oxide coagulation.

Thus, sintered beryllium is a dispersion-strengthened composite material in which beryllium oxide particles play as a reinforcing phase. However, the available large experimental data characterizing the level of properties of industrial and experimental varieties of beryllium does not allow us to identify reliable mathematical models for controlling and increasing mechanical properties to the required level. Insufficient knowledge of the mechanisms and kinetics of reinforcing beryllium oxide particles formation processes complicates the establishment of quantitative dependences of the chemical, granulometric compositions and technological modes, effect on the mechanical properties of products made of beryllium powder.

The aim of the article is to study the mechanism of strengthening beryllium oxide particles formation at grain boundaries in the processes of beryllium powders consolidation and the effect of the reinforcing phase on the sintered beryllium strength properties.

Material and methods of research

Research work aimed at studying the processes of beryllium hardening was divided into two parts. In the first part of the work the formation mechanisms and the reinforcing phase morphology were studied. In the second part of the work the quantitative effect of "dispersion-grain boundary" hardening was studied.

In the first part of the work 32 billets with 380 mm diameter obtained by vacuum hot pressing from different industrial batches of PTB-56 brand beryllium powders were selected as the research material. The workpieces were obtained using standard technology including cold isostatic pressing on a UGS 350 unit designed by VNIIMETMASH followed by vacuum hot pressing on a GPV-200 furnace at 1030°C. Samples for mechanical tests were made from the obtained blanks. Upon mechanical tests completion samples, broken halves that showed significantly different test results in strength properties were selected (Table 1). This selection was examined using a scanning electron microscope with a Jeol ISM 5610 microanalyzer. The morphology, nanostructure and composition of oxide particles were studied at the transboundary fracture areas of the selected samples.

In the second part of the work technical beryllium powders of various fineness (-30, -56, -100, -180 microns) (Figure 1) produced of ingots similar in chemical composition with a low content of low-melting impurities and Si / Al \approx 1 ratio were investigated. This ratio provides the transition of beryllium oxide from the amorphous to the crystalline state (devitrification) by the mechanism of homogeneous crystallization, fine reinforcing particles obtaining the maximum hardening effect [8].

Preparation and hot isostatic pressing of articles made of beryllium powder was carried out according to the standard technology as follows: beryllium powder was poured into elastic molds of 100 mm diameter and 200 mm height and compacted on a UGS 350/1000 hydrostat at 4 kbar pressure. After uncovering the porous briquettes were placed in steel sealed capsules, degassed in vacuum at 700°C and subjected to hot isostatic pressing in the temperature range 800–1100°C to 1.85 g/cm³ density. Samples with 5 mm diameter cut from compact blanks were tensile tested on Instron 1195 testing machine according to well-known methods which made it possible to determine the conventional yield stress $\sigma_{0.2}$ and the precision elastic limit $\sigma_{0.005}$. The microstructure was studied with an optical microscope, and the dispersion of Beryllium oxide at the grain boundaries was studied with a transmission electron microscope using replicas obtained from samples, fractures [11].

Results and discussions

1. Study of the formation mechanism and morphology of the reinforcing phase

Table 1

Characteristics of samples

Preform №	Content of main impurities, % mass				Average grain size, μm	Ultimate strength, MPa
	Oxygen	Silicon	Aluminum	Iron		
1	0.8	0.023	0.023	0.12	41	337
2	0.75	0.012	0.027	0.13	26	405

Note: 1) The tensile strength values were obtained by averaging over the results of three tests;
2) Conditioned products shall have a tensile strength of more than 350 MPa

Micro-fractograms analysis of samples fractures from preforms No. 1 and No. 2 showed that structural differences consist not only in the grain size, but also in the morphology and structure of the strengthening particles of the oxide phase (Figure 1).

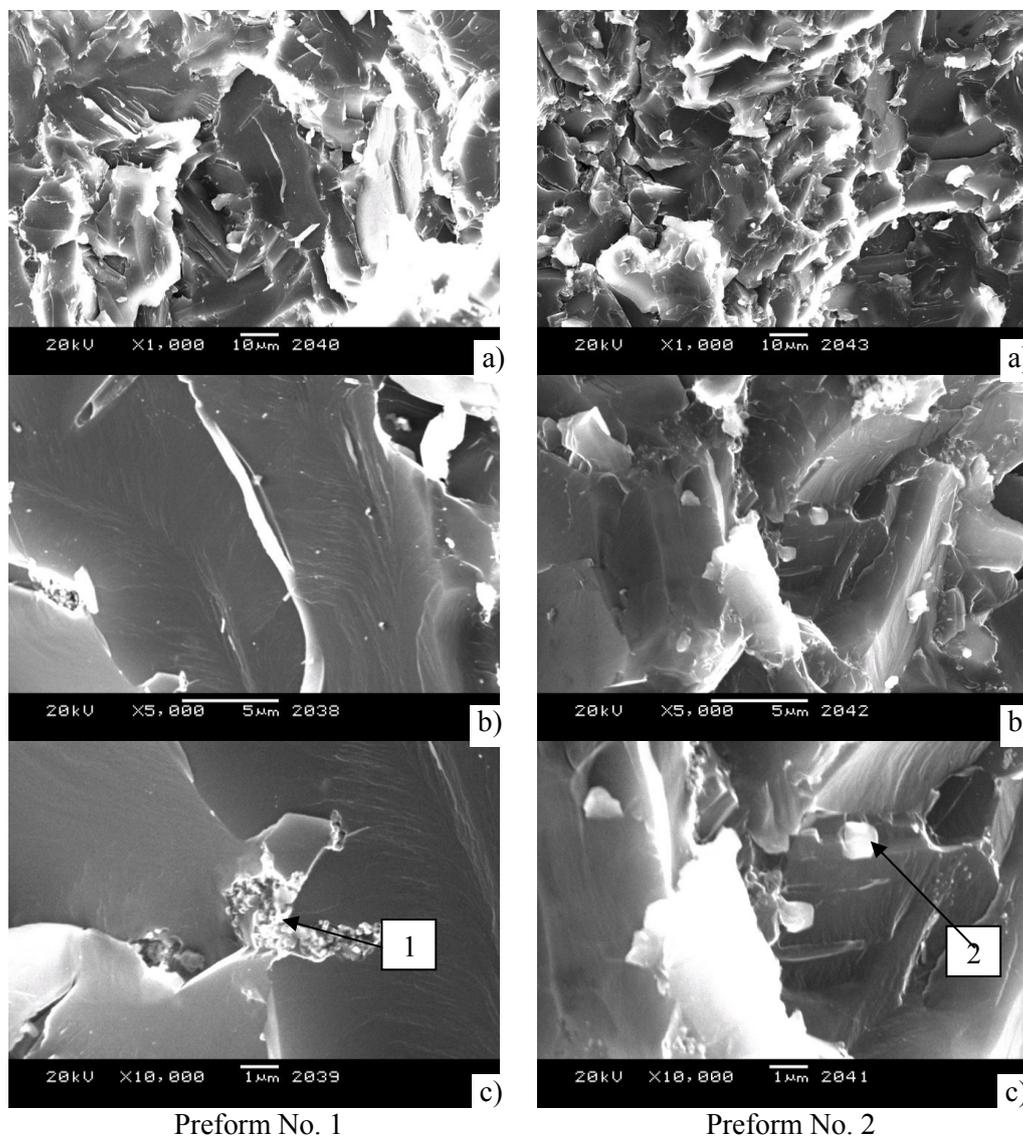


Figure 1. Microfractography of fractures of hot-pressed Beryllium PTB-56

Preform No. 1: sample with Si content = 0.023%. a) $\times 1000$; b) $\times 5000$; c) $\times 10000$.
Preform No. 2: sample with Si content = 0.012%. a) $\times 1000$; b) $\times 5000$; c) $\times 10000$.

With the same amounts of beryllium oxide in the preforms and identical pressing modes, fine particles of single-crystal beryllium oxide are formed in preform No. 2 (Si content = 0.012%), and large polycrystalline clusters consisting of nanoparticles oxide cemented with a metal bond based on Si and Al are formed in preform No. 1 (Si content = 0.023%). These structural and morphological differences determine a different strengthening effect of beryllium: the reinforcing phase in the form of small single crystals of beryllium oxide causes a greater strengthening effect than large polycrystalline clusters (such as cermets) (see Table 1).

The differences in the morphology, composition and structure of reinforcing particles can be explained by the fact that the formation of oxide particles at the grain boundaries of sintered beryllium is the result of non-cracking of the oxide film under the action of stresses caused by the difference in the temperature expansion coefficients of the metal and oxide [3, 6], and the transition of amorphous beryllium oxide into a crystalline state, the so-called devitrification. Moreover, this transition, as shown by further studies, can take place at the mechanisms of homogeneous and heterogeneous crystallization [8–10].

The homogeneous mechanism of beryllium oxide devitrification at the grain boundaries of sintered beryllium provides a high strengthening effect both due to the formation of a highly dispersed reinforcing phase and due to the finer grain size of metallic beryllium microstructure (Figure 2 a).

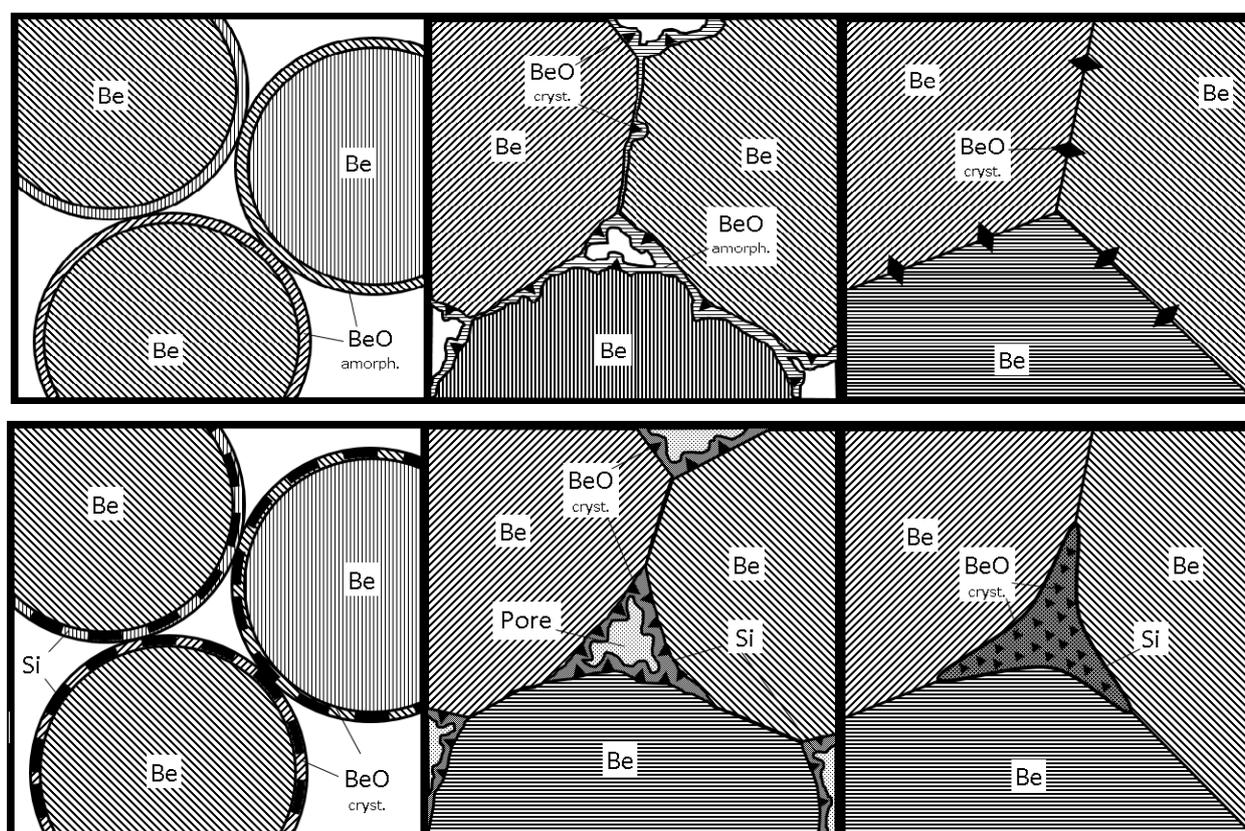


Figure 2. Formation of the structure of Beryllium oxide reinforcing phase at the grain boundaries of sintered Beryllium at various contents of silicon impurities

- a) The transition of an oxide from an amorphous state to a crystalline state by the mechanism of homogeneous crystallization;
- b) The transition of an oxide from an amorphous state to a crystalline state by the mechanism of heterogeneous crystallization;

The heterogeneous mechanism of beryllium oxide devitrification caused by unfavorable ratio of silicon to aluminum impurities induces a decrease in the strengthening effect both due to the formation of large oxide-silicon-aluminum clusters during oxide devitrification and due to the significant growth of beryllium grains (Figure 2 b). The above is a feature of the "dispersed-grain-boundary" hardening mechanism of sintered beryllium. Analysis of the results obtained after statistical processing shows that, in addition to the total

amount of impurities forming the low-melting Si + Al + Mg eutectic, the powder particle size and the Si/Al impurity ratio are significant. Even greater reliability of strength properties prediction is achieved when both parameters $d \times (\text{Si} + \text{Al} + \text{Mg}) \times \text{Si}/\text{Al}$ are taken into account (Figure 3).

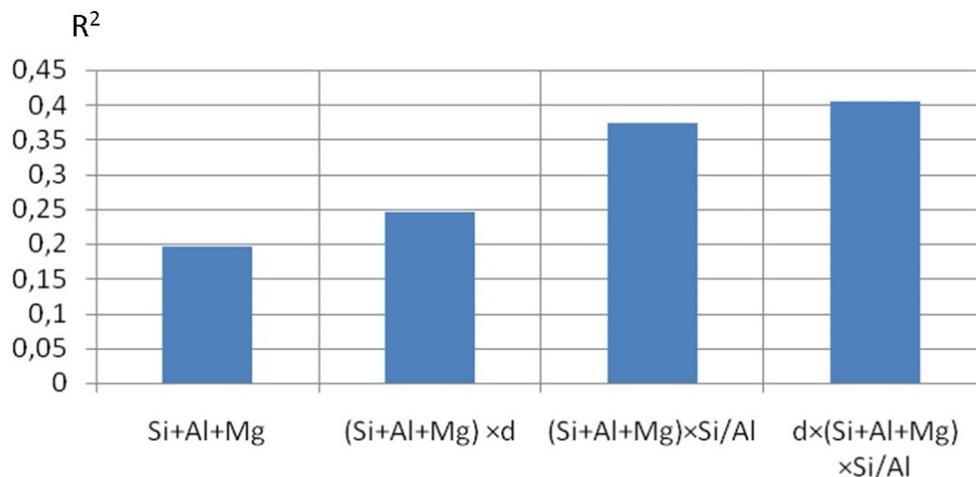


Figure 3. Dependence of the approximation reliability coefficient on quality indicators of initial powders

2. Study of the quantitative effect of "dispersed grain boundary" hardening

Microfractography results analysis of isostatically pressed PTB 56 grade beryllium samples fractures with an electron microscope showed that the beryllium oxide film on the hereditary grain boundaries in the samples pressed at 800 and 900°C temperatures practically did not collapse (Figure 4).

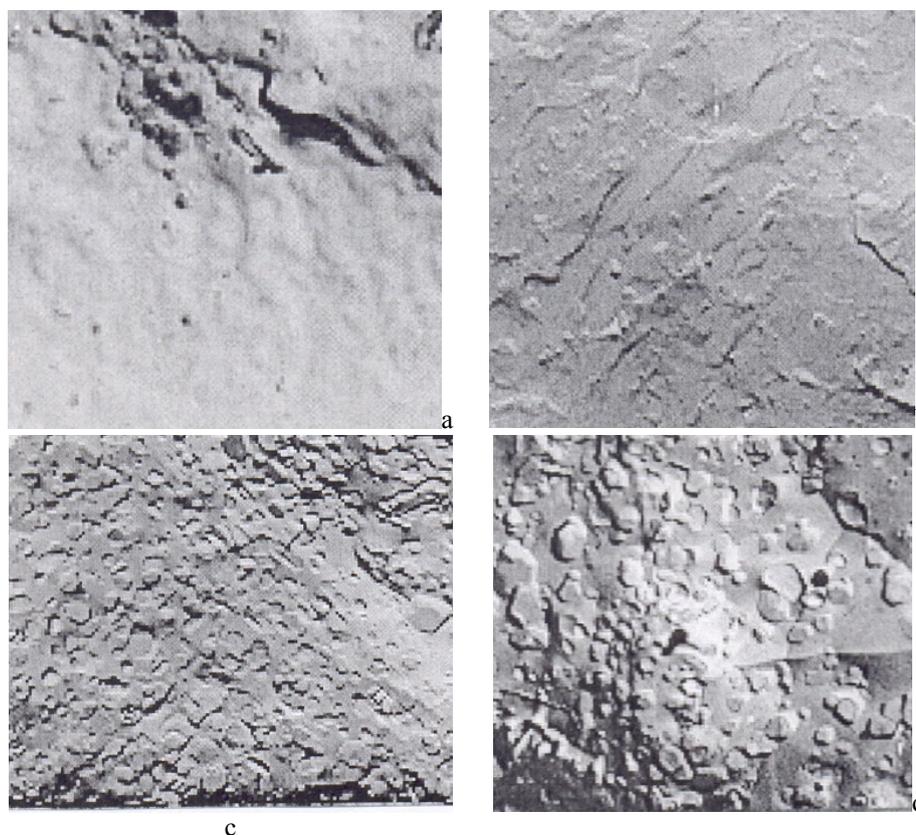


Figure 4. Fractogram of isostatically pressed grade PTB-56 Beryllium samples fractures at 800 (a), 900 (b), 1000 (c) and 1100 ° C (d), × 10000.

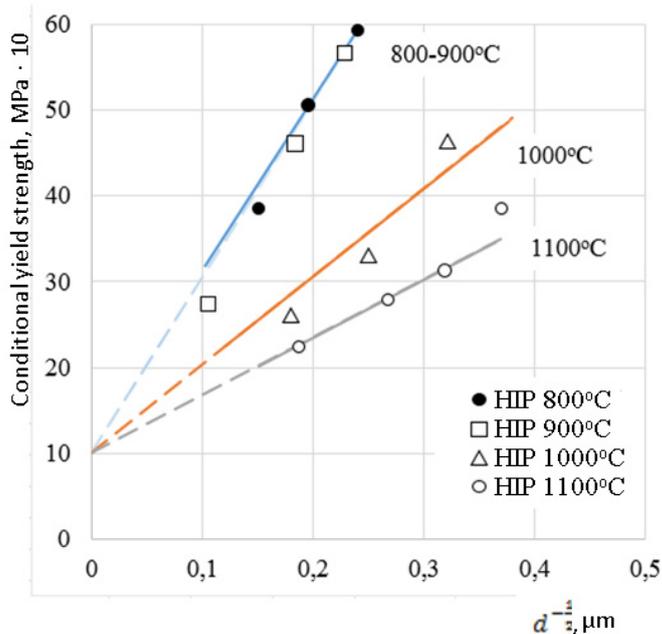


Figure 5. Dependence of the conventional yield strength on grain d size (Hall-Petch dependence) and temperature of isostatic pressing

Herewith, local thickenings are observed on the oxide film, which are nuclei of crystalline beryllium oxide formed as a result of homogeneous devitrification of the initially amorphous form of the oxide. The average size of the thickenings is 6 and 10 nm, respectively. An increase in the pressing temperature to 1000 and 1100°C causes discrete beryllium oxide reinforcing particles formation with an average size of 120-150 and 300-400 nm, respectively. Dependence of the preforms strength properties on the average grain size and the temperature of isostatic pressing were found. (Figure 5).

These graphical dependencies are consistent with the Hall-Petch equation as well as with the Orowan mechanism [12] where dislocation loops are formed around the inclusions when dislocations pass through an obstacle (Figure 6).

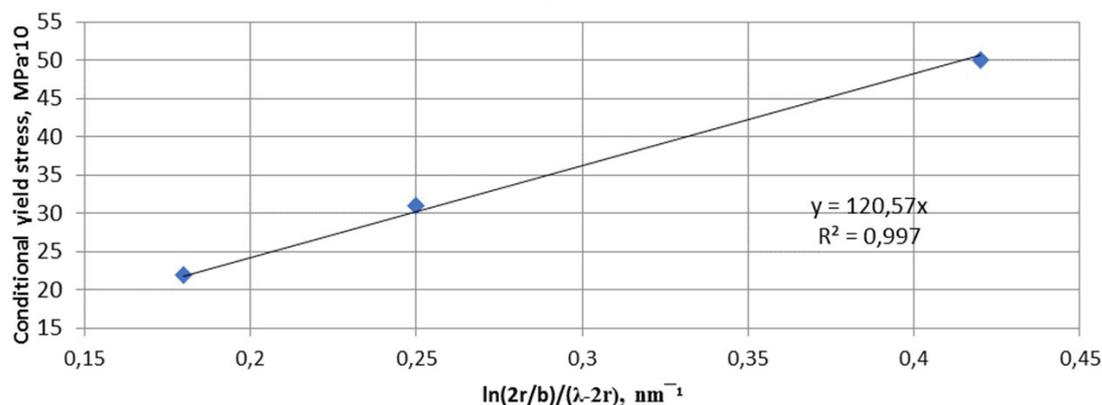


Figure 6. Dependence of the increment of the conventional yield stress on the size factor marking the dispersion of oxide particles at a constant grain size ($d^{-1/2}$) in the microstructure of the preforms; r is the radius of the particle, b is the Burgers vector of Beryllium, (λ) is the distance between the particles

Thus, the Beryllium structure formation in the process of hot isostatic pressing of its powders coated with an oxide film is accompanied by hardening, which is a superposition of grain boundary and disperse mechanisms. The conventional yield stress of Beryllium obtained from powders of various sizes and isostatically compressed at different temperatures is related to the precision tensile strength in a complex way (Figure 7).

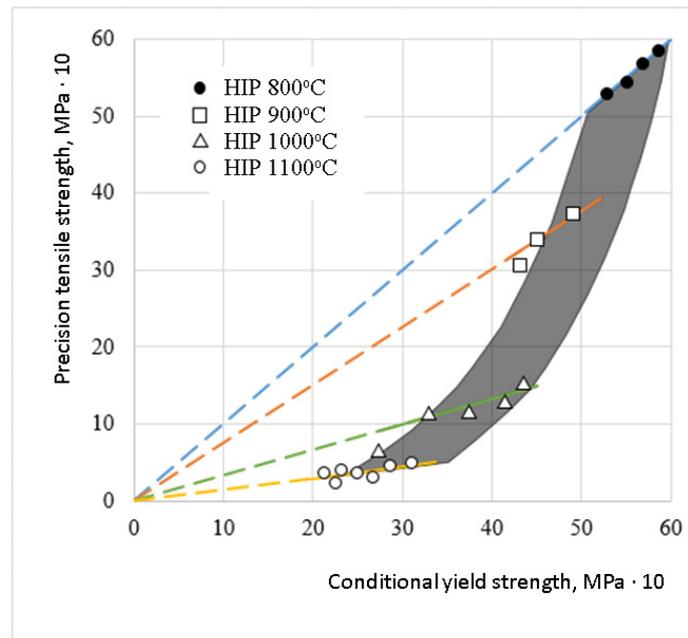


Figure 7. The dependence of the precision tensile strength on the yield strength

The experimental results obtained can be approximated in the form of analytical dependences of the precision tensile strength on the beryllium grain size, beryllium oxide particle size (1), and hot isostatic pressing temperature (2):

$$\sigma_{0.005} = a(100 + m d_0^{-0.8} \cdot d^{-0.5})^4, \text{MPa}, \tag{1}$$

where a , m are coefficients equal to $6 \cdot 10^{-9}$, $3.7 \cdot 10^3$, respectively; d_0 is the average size of oxide particles, nm; d is the average grain size of beryllium, microns;

$$\sigma_{0.005} = a(100 + m t_{HIP}^{-4} \cdot d^{-0.5})^4, \text{MPa}, \tag{2}$$

where a , m are coefficients equal to $6 \cdot 10^{-9}$, $1.07 \cdot 10^{15}$, respectively; t_{HIP} is the temperature of hot isostatic pressing; d_0 is the average grain size of beryllium in microns.

The part of the equation in brackets is essentially the Hall-Petch equation for beryllium obtained by hot isostatic pressing with coefficients at $d^{-0.5}$ equal to $m d_0^{-0.8}$ and $m t_{HIP}^{-4}$ when substituting for the oxide particle size d_0 or the hot temperature isostatic pressing t_{HIP} , respectively. The indicated dependence reflects the additive contribution of grain boundary and dispersed strengthening mechanisms.

Thus, the results of the study reflect the regularities of polycrystalline beryllium hardening by a mechanism that is a superposition of two mechanisms: grain boundary and disperse hardening. An analysis of the dependences shows that these mechanisms almost equally affect the values of the conditional yield stress, at the same time, the precision tensile strength significantly depends on the state of the oxide and weakly depends on the grain size. This is apparently due to the fact that the level of the precision tensile strength is controlled by the mechanism of grain-boundary dislocations generation blocked by fine particles of dispersed beryllium oxide, which leads to an increase in the precision tensile strength. The conditional yield point is controlled mainly by the grain-boundary hardening mechanism, i.e., the mean free path of dislocations from one grain boundary to another. Using beryllium powders of the same dispersion, changing only the dispersion of the strengthening phase in the range of 6–400 nm, it is possible to change the strength properties in a wide range, for example, a material with a precision tensile strength can be obtained from PTB 56 powder in the range of 50–500 MPa.

The established graphical and analytical dependencies open up the possibilities of fine control of the strength properties of beryllium by dispensing the contribution of each of the elements of the "dispersed-grain-boundary" hardening mechanism.

Conclusion

According to the results of the first part of the work during the study of the reinforcing phase formation it was found that the degradation of the oxide film on the surface of beryllium grains during heating proceeds according to the devitrification mechanism: the transition from the amorphous state to the crystalline state, and it was shown that the strengthening effect of sintered beryllium obtained by hot molding is largely determined by the mechanism of beryllium oxide devitrification at the grain boundaries of metallic beryllium. The devitrification process, morphology and nanostructure of reinforcing oxide particles are affected by low-melting impurities, in particular silicon and aluminum, and depending on the content and ratio of silicon and aluminum impurities this mechanism can be either homogeneous or heterogeneous. Depending on by which devitrification mechanism oxide particles are formed, they cause effective strengthening (homogeneous mechanism) and weak strengthening (heterogeneous mechanism), this determines the morphology, composition and structure of the reinforcing phase as well as the grain size of metallic beryllium that ultimately determines the strengthening effect according to the "dispersion-grain-boundary" mechanism.

According to the results of the second part of the work it is shown that the level of the precision limit of isostatically compressed beryllium elasticity is controlled by the "dispersed-grain-boundary" mechanism. The quantitative dependences of the precision elastic limit of isostatically compressed beryllium on the heating temperature, the size of the beryllium grain, and the size of the reinforcing oxide particles was established. Thus, using beryllium powders of the same dispersion changing only the dispersion of the strengthening phase in the range of 6–400 nm it is possible to change the strength properties in a wide range, for example, a material with a precision elastic limit can be obtained from PTB 56 powder in the range of 50–500 MPa.

The established graphical and analytical dependencies open up possibilities for fine control of beryllium strength properties by dosing the contribution of each element of the "dispersed-grain-boundary" hardening mechanism.

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Ұнтақты металлургия әдісімен алынған бериллийді беріктендіруді зерттеу

Авторлар мақаланың бірінші бөлімінде бериллий ұнтақтарын ыстық престоу әдісімен консолидациялау кезінде пайда болатын ұнтақты беріктендіру процестерін зерттеудің нәтижелерін көрсеткен. Беріктендіргіш фазасының құрамы мен морфологиясының агломерленген бериллий ұнтағы түйіршіктерінің бетіндегі оңайбалқығыш қоспалардың құрамына тәуелділігі зерттелді. Оксид пленкасының аморфтыдан кристалдық күйге ауысуын – ыдырау процесі механизміне оңайбалқытын қоспалардың әсерін және «дисперсті-ұнтақты» эффектіндегі беріктендірудің әсері гипотезасы арқылы түсіндіріледі. Бұл гипотезаның негізі дисперсті-ұнтақты беріктендіру механизмімен бірге ыдырау процесін көрсететін графикалық материалды ұсынумен теориялық растау болып табылады. Қоспалар құрамының ыстық престелген бериллийдің қасиеттеріне тәуелділігін көрсететін өндірістік партиялар бойынша жүргізілген статистикалық өңдеудің соңғы нәтижелері келтірілген. Жұмыстың екінші бөлімінде ұнтақтардың шоғырлану температурасына байланысты ыстық изостатикалық престоу (БИП) әдісімен агломерация процесінде алынған бериллийді беріктендіру әсерінің зерттеу нәтижелері берілген. Электронды микроскопиялық зерттеулердің нәтижелері бойынша агломерленген бериллий түйіршіктерінің шекарасында арматуралық фазаның түзілу динамикасы көрсетілген. Дәлдік серпімділік шегі мен газстатикалық престелген бериллийдің шартты аққыштық шегі арасындағы сандық тәуелділігі бериллий оксиді беріктендіргіш бөлшектерінің мөлшеріне және ұнтақтардың шоғырлану температурасына байланысты. Алынған теңдеу изостатикалық престелген бериллийдің «дисперсті-түйіршіктішекарасы» беріктену механизміне сипаттама береді. Жүргізілген зерттеудің мәнін бейнелейтін графикалық материалдардың деректер тәуелділігі берілген.

Кілт сөздер: бериллий, ұнтақ, ыстық изостатикалық престоу, микроқұрылым, бериллий оксиді, престоу температурасы, электронды микроскопия, механикалық қасиеттер, дисперстілігі, серпімділік шегі, аққыштық шегі.

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Исследование упрочнения бериллия, полученного методом порошковой металлургии

Авторами первой части статьи показаны результаты исследования процессов упрочнения порошка, происходящих при консолидации порошков бериллия, методом горячего прессования. Изучены зависимости содержания и морфологии упрочняющей фазы в зависимости от содержания легкоплавких примесей на поверхности зерен спеченного порошка бериллия. Предложена гипотеза, объясняющая факт перехода оксидной пленки из аморфного в кристаллическое состояние, – расстеклование и влияние легкоплавких примесей на механизм процесса расстеклования, и, как следствие, на эффект «дисперсно-зернограничного» упрочнения. В основе данной гипотезы лежит теоретическое подтверждение с предоставлением графического материала, демонстрирующего процесс расстеклования, сопровождающийся дисперсно-зернограничным механизмом упрочнения. Приведены итоговые результаты статистической обработки, проведенной на промышленных партиях, показывающие зависимости влияния содержания примесей на свойства горячепрессованного бериллия. Во второй части работы представлены результаты исследования эффекта упрочнения бериллия, полученного в процессе спекания методом горячего изостатического прессования (ГИП), в зависимости от температуры консолидации порошков. На основании данных электронно-микроскопических исследований показана динамика формирования армирующей фазы на границах зерен спеченного бериллия. Установлена количественная зависимость прецизионного предела упругости и условного предела текучести газостатически спрессованного бериллия от размера упрочняющих частиц оксида бериллия и температуры консолидации порошков. Полученное уравнение представляет собой описание «дисперсно-зернограничного» механизма упрочнения изостатически спрессованного бериллия. Все зависимости даны также графическим материалом, отражающим суть проведенных исследований.

Ключевые слова: бериллий, порошок, горячее изостатическое прессование, микроструктура, оксид бериллия, температура прессования, электронная микроскопия, механические свойства, дисперсность, предел упругости, предел текучести.

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