DOI 10.1007/s11182-020-01901-2

Russian Physics Journal, Vol. 62, No. 9, January, 2020 (Russian Original No. 9, September, 2019)

BRIEF COMMUNICATIONS

MICROSTRUCTURE AND MICROHARDNESS OF A MULTICOMPONENT SYSTEM AFTER MECHANICAL ACTIVATION AND SPARK PLASMA SINTERING

I. A. Ditenberg,[1,2] M. A. Korchagin,[3] I. V. Smirnov,[1,2] K. V. Grinyaev,[1,2] Yu. P. Pinzhin,[1,2] V. V. Melnikov,[1] D. A. Osipov,[1] A. I. Gavrilov,[3] M. A. Esikov,[4] and V. I. Mali[4]

UDC 538.911; 548.4; 669.17: 620.186.8

Keywords: mechanical activation, spark plasma sintering, multicomponent system.

Multicomponent systems based on refractory metals are of practical interest for applications in nuclear power generating and aerospace industries [1–5]. The investigations in these fields are primarily focused on improving the thermal stability of strength properties of materials at high temperatures. A problem of great concern in metallurgy during production of such systems is a considerable difference in the melting temperatures of their components (e.g., W – 3380°C, Ta – 3014°C, V – 1920°C, Ti – 1671°C), due to which the traditional approaches turn out to be unviable. One of the methods for manufacturing such systems is the use of mechanical activation (MA) [6] followed by spark plasma sintering/synthesis (SPS) [7, 8]. This approach has been successfully used for the synthesis of intermetallic compounds [9] and high-entropy alloys of various systems [5, 10–12].

In the present work we investigate microstructure and microhardness of a multicomponent alloy based on refractory metals (W–Ta–Mo–Nb–V–Cr–Zr–Ti), which was manufactured by mechanical activation of a powder mixture and subsequent spark plasma sintering.

The elemental composition of this system in weight and atomic percent is presented in Table 1.

The mechanical activation was performed in an AGO-2 water-cooled planetary ball mill. The volume of every of the two steel vials of the mill is 160 см^3, the ball diameter – 8 mm, the ball mass in every vial – 200 g, the specimen weight – 11.44 g. The centrifugal acceleration of the balls is 400 m/s^2 (40g). In order to prevent oxidation, the specimens were processed and unloaded in an argon atmosphere. The MA duration in this regime was 10.5 min. Aggregation of the powder mixture before unloading was prevented by its processing (up to 0.5 min) with added ethanol. The spark plasma sintering was performed in an SPS Labox-1575 system at a pressure of 40 MPa at the temperature 1250°C and holding time no less than 5 min. The specimens were produced in the shape of tablets measuring 20 mm in diameter and 1.5 mm in thickness. Their Vickers microhardness (H_μ) was determined in a Neophot-21 metallurgical microscope at a load of 0.5 N and an exposure of 15 s.

[1]National Research Tomsk State University, Tomsk, Russia, e-mail: pinzhin@phys.tsu.ru; melnikov@phys.tsu.ru; osipov_ff_tsu@mail.ru; [2]Institute of Strength Physics and Materials Science of the Siberian Branch of the Russian Academy of Sciences, Tomsk, Russia, e-mail: ditenberg_i@mail.ru; smirnov_iv@bk.ru; kvgrinyaev@inbox.ru; [3]Institute of Solid State Chemistry and Mechanochemistry of the Siberian Branch of the Russian Academy of Sciences, Novosibirsk, Russia, e-mail: korchag@solid.nsc.ru; [4]Lavrentiev Institute of Hydrodynamics of the Siberian Branch of the Russian Academy of Sciences, Novosibirsk, Russia, e-mail: esikov@hydro.nsc.ru; mali@hydro.nsc.ru. Translated from Izvestiya Vysshikh Uchebnykh Zavedenii, Fizika, No. 9, pp. 185–186, September, 2019. Original article submitted May 30, 2019.

TABLE 1. Elemental Composition of a Powder System in Weight (wt.%) and Atomic (at.%) Percent

Content	W	Ta	Mo	Nb	V	Cr	Zr	Ti
Wt. %	12.5	12.5	12.5	12.5	12.5	12.5	12.5	12.5
At. %	5.3	5.4	10.1	10.5	19.1	18.7	10.7	20.3

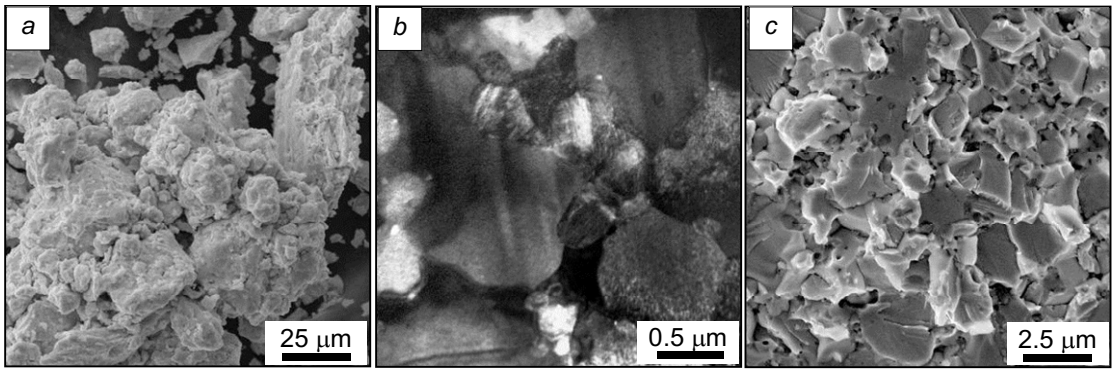

Fig. 1. Structure of a multicomponent system in different stages of its processing. Precursor morphology after MA (*a*), sintered specimen microstructure (*b*), fracture photomicrograph (*c*) obtained at a temperature of about –196°C. Scanning (*a*, *c*) and transmission (*b*) electron microscopy.

The microstructure was examined by the method of transmission electron microscopy in a Philips CM-12 (120 kV) microscope. The powder mixture morphology after MA, the energy dispersive analysis of the chemical composition of the precursors and synthesized workpieces were investigated using the FEI Quanta 200 3D (30 kV) and Tescan Vega 3 SBH (20 kV) scanning electron microscopes.

The precursor formed by MA is characterized by the presence of conglomerates from a few tens to hundreds of micrometers (Fig. 1*a*) consisting of fine (submicron and micron dimensions) powder particles of the initial components. The results of the energy dispersive analysis suggest a homogenous mechanical mixing of the multi-component system in the MA stage.

The sintered specimen microstructure consists of the grains of submicron and micron dimensions (Fig. 1*b*) differing in their phase compositions. Inside certain grains, the defect structure is present in the form of dislocations and twins; also there are second-phase inclusions. The fracture photomicrograph of the specimen cross section (Fig. 1*c*) obtained at the liquid nitrogen temperature is characterized by the brittle fracture type with spalling of the finest crystallites.

Following MA, the precursor microhardness is characterized by a strong inhomogeneity: at the average value 9.28 GPa the scatter is found to be ±1.31 GPa. As a result of SPS, the average microhardness value decreases to 8.95 GPa and the deviations are not higher than ±0.42 GPa.

Using a W–Ta–Mo–Nb–V–Cr–Zr–Ti model system, we have demonstrated a possibility of using MA followed by SPS for manufacturing bulk specimens from a multicomponent system based on refractory metals at the temperature 1250°C. Generally in order to produce similar systems, consisting of refractory metals only, sintering is performed at the temperature no less than 1400°C [5, 12]. The formation of a precursor in the MA stage, in which the micron and submicron particles of the initial components are distributed homogeneously, results in a grain structure with comparable dimensions in the specimens consolidated after sintering. Its multicomponent composition, consisting of the elements dissolving each other, ensures a possibility of forming a wide range of solid solutions, which favors the creation of a multiphase structural state under the solid-phase synthesis conditions.

The structural studies were performed using the equipment of the Tomsk Materials Center for Shared Use of Scientific Equipment at NR TSU.

This work was carried out using the support of the Ministry of education and science of the Russian Federation (Project No. 3.9586.2017/8.9).

REFERENCES

1. D. B. Miracle and O. N. Senkov, Acta Mater., **122**, 448–511 (2017).
2. X. Xian, Z. Zhong, B. Zhang, *et al*., Mater. Design., **121**, 229–236 (2017).
3. O. N. Senkov, G. B. Wilks, D. B. Miracle, *et al*., Intermetallics, **18**, No. 9, 1758–1765 (2010).
4. A. M. Manzoni, S. Singh, and H. M. Daoud, Entropy, **18**, No. 104, 1–13 (2016).
5. J. Yan, K. Li, Y. Wang, and J. Qiu, JOM, **71**, Iss. 8, 2489–2497 (2019).
6. C. Suryanarayana, Prog. Mater. Sci., **46**, No. 1–2, 1–184 (2001).
7. M. Tokita, Mater. Sci. Forum, **308**–**311**, 83–88 (1999).
8. Spark Plasma Sintering of Materials (Ed. P. Cavaliere), Springer Nature Switzerland AG, Cham (2019).
9. L. I. Shevtsova, M. A. Korchagin, A. Thömmes, *et al*., Adv. Mater. Res., **1040**, 772–777 (2014).
10. A. Kumar, A. K. Swarnakar, and M. Chopkar, J. Mat. Eng. and Perform., **27**, Iss. 7, 3304–3314 (2018).
11. M. Vaidya, G. M. Muralikrishna, and B. S. Murty, JMR, **34**, No. 5, 664–686 (2019).
12. Y. Long, X. Liang, K. Su, *et al*., J. Alloys Compounds, **780**, 607–617 (2019).