OBTAINING OF Nb-16Si SPHERICAL POWDERS ALLOY FOR ADDITIVE TECHNOLOGIES BY MECHANICAL ALLOYING AND SPHEROIDIZATION IN ELECTRIC ARC DISCHARGE THERMAL PLASMA

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ABSTRACT

Nb-16Si (at. %) powder alloy was prepared by mechanical alloying (MA) in a planetary ball mill (Fritsch Pulverisette-4) from pure elemental powders. For the process of Nb-16Si powder spheroidization an experimental plasma generator based on thermal plasma arc generator with vortex discharge stabilization was used. Experimental results show that Nb-16Si spherical powder with a high degree of spheronization using starting powder fractions of +45-71 µm and +71-100 µm can be prepared in a stream of argon thermal plasma with hydrogen additives generated in plasmatron. It is shown that the main peaks in the X-ray graph after MA correspond to a solid solution of niobium with a cubic lattice and the parameter a = 0.333 nm, as well as niobium silicide Nb5Si3 with a hexagonal lattice (P63/m) a = 0.7536 nm and c = 0.5249 nm. After spheroidization the hexagonal lattice of niobium silicide Nb5Si3 is transformed into a tetragonal lattice (I4/m) with the parameter a = 0.6557 nm and c = 1.186 nm. The other phase components remain unchanged.

Keywords: mechanical alloying, Nb-Si, spheroidization, thermal plasma, spherical powder.

INTRODUCTION

Additive technology (AT), commonly known as three-dimensional printing, is a method of making products of various shapes by adding layers of material in powder, wire, fluid, etc, using a computer model [1]. Three-dimensional printing of products from metals and their alloys is of great interest for many high-tech industries. Enhanced interest in AT is caused primarily by its ability to create objects of complex geometric shapes within a short period of time without the need for additional manufacturing equipment [2].

The general requirement to powders for additive technology is a spherical particle shape and particle size distribution of high uniformity. The spherical shape provides a more compact packing of particles in certain volumes and fluidity of the powder with a minimum resistance in the material feed systems. Today, the characteristics of powders limit the field of application of additive technologies, e.g. a narrow range of powders is produced due to their chemical composition. As a result, it is impossible to provide necessary structural and functional properties of materials, neither to obtain high manufacturing precision and surface quality [2].

Over 90% of all powders used in additive technologies are obtained by melt dispersion methods. Main technologies for the producing powders for layered synthesis units include gas atomization and centrifugal atomization [3, 4]. Gas atomization method allows obtaining fine powders with the particles of spherical shape, guarantees high productivity of the process with a controlled dispersion of powders and provides a high yield of fractions for powder. However, the method has several disadvantages. For example, the powder particles produced by gas atomization may have internal porosity due to inert gas collapsing inside the particles during crystallization. The most typical and inevitable defect of the powders obtained by spraying liquid metal with the flow of inert gas is satellite formation, that is "sticking" of small particles onto larger ones. Unlike centrifugal atomization, in which granules are sprayed at 360 degrees, a jet formed during gas atomization is directed along the axis of atomization column to maximize atomization "departure" of the particles without the contact with the walls to maintain the sphericity. Under such conditions the satellites formation is unavoidable, since the energy of gas stream is transferred to the particles depending on their weight: the most dispersible granules acquire a high initial rate and, crystallizing instantaneously, bombard larger partially crystallized pellets with lower initial velocity.

The alternative methods of producing powders are mechanical methods, such as grinding and mechanical alloying [5]. Now, powders obtained by mechanical alloying are not used for additive technologies, since powder particles are of fragmentation irregular shape. For powders spheronization different methods of spheroidization are used. There are several methods of powders spheroidization, one of which uses thermal effects of the plasma stream. At a high temperature, reaching 10000°C in the plasma jet, it becomes possible to melt and evaporate even the most refractory compounds. By controlling such parameters as a flow rate, powder trajectory, plasma gas flow and plasma flow capacity, possible obtain the optimum mode of producing particles of a spherical form in the compounds of the given composition [6-10].

The paper presents the results of experimental research carried out while processing the powder of Nb-16Si alloy (at.%) produced by mechanical alloying of

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elemental powders of Nb and Si in the flow of a thermal plasma.

MATERIALS AND METHODS

Nb-16Si (at. %) powder alloy ware prepared by mechanical alloying of pure elemental powders in the Fritsch Pulverisette 4 planetary mill. High purity Nb (d90 < 200 μm, 99.96% purity; Huizhou Gl Technology Co., LTD, PRC) and Si (d90 < 100 μm, 99.95% purity; Neva Reaktiv, LLC, Russia) were used as initial components. To prevent oxidation of the initially-formed powder, all operations were handled inside a glove box under a high-purity argon atmosphere. Powders initially nominally composed of Nb and 16 at.% Si were milled for 6 h using a planetary ball mill with hard metal vials and balls under an argon atmosphere and a ball-to-powder mass ratio of 10:1 using a disc rotation velocity of 200 rpm. The main-to-planetary disc rotation velocity ratio was 1:-1.5.

For the process of Nb-16Si powder spheroidization a plasma generator based on thermal plasma arc generator with the vortex discharge stabilization was used. Spheroidization of metal powders in plasma unit is based on heating and melting the initial metallic particles introduced into the plasma jet by carrier gas. Upon cooling the high-temperature gas-dispersed flow in a reactor with water-cooled walls metal particles in the form of spheres are crystallized. The resulting spherical powder is deposited on the inner walls of the reactor in its conical bottom, partly imposed on the bag filter, and collected into a hopper of the expected product. The experimental studies of Nb-16Si powder spheroidization in the flow of thermal plasma generated in an arc-jet plasmatron were carried out within torch net power - 7 kW and plasma gas flow - 2 m^3/h.

The phase composition was studied by X-ray analysis on Brucker D8 Advance unit in CuKα-radiation (λ = 1,5418Å). Further processing of the diffraction data was carried out by Rietveld method using Diffrac Plus Topas software produced by BRUKER. Phase composition was determined by comparing the set-spacings of experimentally obtained radiographs with the radiometric data base powder phase PDF-2 (ISDD). The morphology of the particles and the structure of the obtained alloys were studied using Scios scanning electron microscope (FEI Company) with the possibility of the elemental analysis (EDX) and layered ion etching (FIB column with a gallium gun).

RESULTS AND DISCUSSIONS

During mechanical alloying of the powders of raw components, Nb-16Si alloy powder of the fragmentation form with 100 μm particle size was obtained (Figure-1). The process of the layer etching of the particles by gallium ions found that the particles are agglomerates of micron and submicron particles.

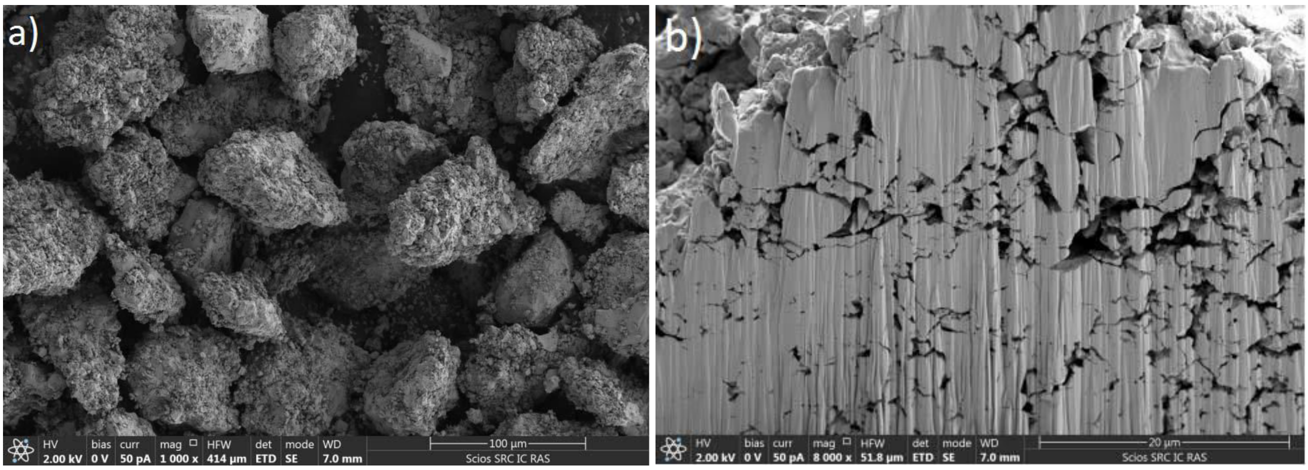


Figure-1. The morphology of Nb-16Si powder particles after mechanical alloying.

The experimental studies revealed that Nb-16Si spherical powder with a high degree of spheroidiization using starting powder fractions of +45-71 μm and +71-100 μm can be prepared in a stream of argon thermal plasma with hydrogen additives generated in plasmatron (Figure 2, 3). In the view of the wide distribution of the particle size and their low mechanical strength, which can lead to their degradation into smaller fragments during plasma spheroidization, the evaporation of the smallest particles followed by the condensation of vapor in the form of nanoparticles can be expected (Figure-3b).

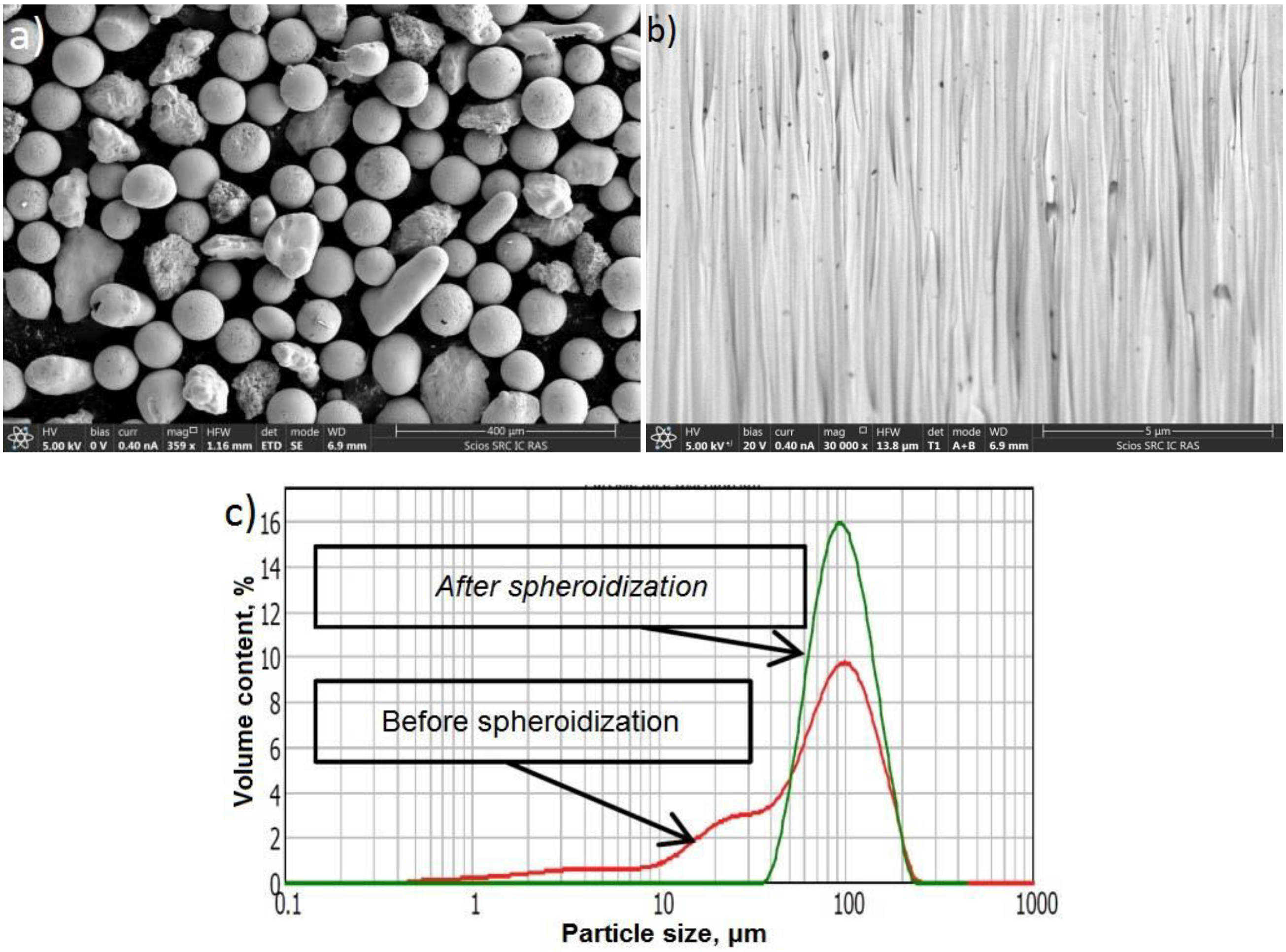
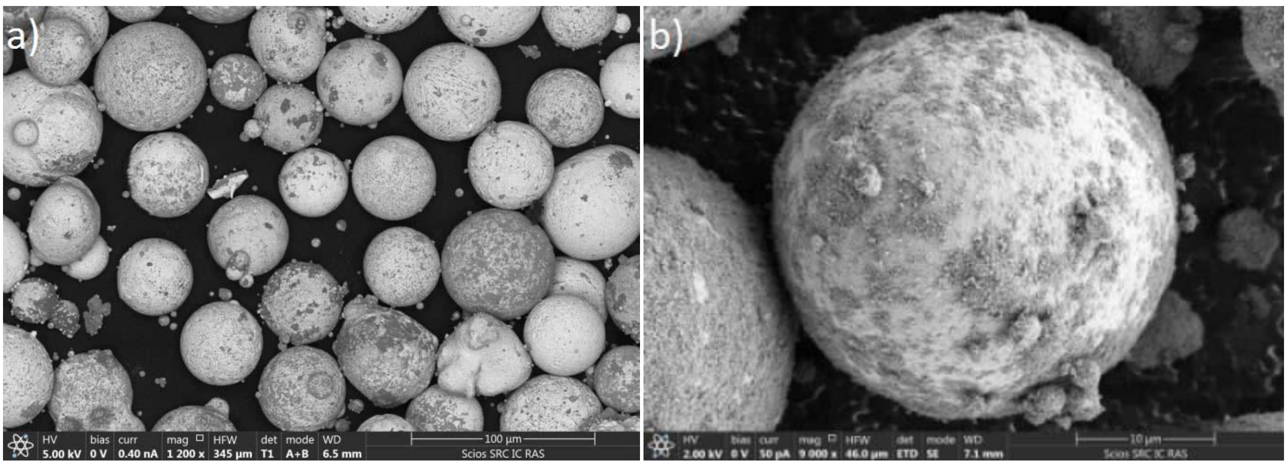


Figure-2. The morphology (a,b) and particle size distribution (c) of Nb-16Si powder of +71-100 µm fraction.



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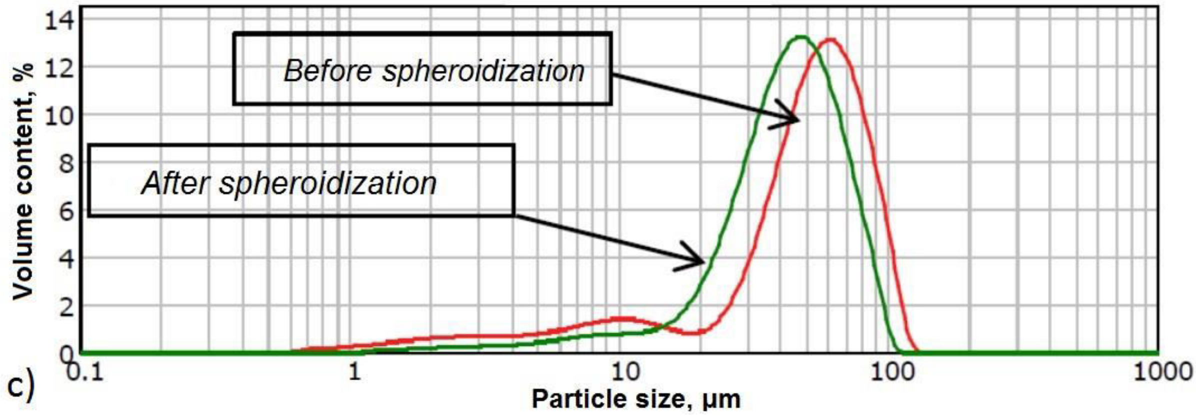

Figure-3. The morphology (a, b) and particle size distribution (c) of Nb-16Si powder of +45-71 μm fraction after plasma spheroidization.

The results of the study into size distribution (Figure 2c and 3c) show that spheroidization leads to particle size reduction due to reflowing and densification of the agglomerates. By the results of layer etching of spheroidized particles by gallium ions (Figure-2a) it was revealed that there are submicron pores developed in the internal structure of the spherical particles. This may be due either to an insufficient degree of penetration of the highly porous particles or to water vapor released as a result of hydrogen reduction of metal oxides.

XRD analysis of the powders obtained by mechanical alloying showed the presence of peaks of the solid solution of niobium (Nb_{SS}) and silicides of niobium - Nb_5Si_3, Nb_3Si (Figure-4). The main peaks in the X-ray graph correspond to a solid solution of niobium with a cubic lattice and the parameter a = 0.333 nm, as well as niobium silicide Nb_5Si_3 with a hexagonal lattice (P63/m) a = 0.7536 nm and c = 0.5249 nm. Broad peaks indicate a strong distortion of the crystal lattice, formed as a result of the intensive mechanical action. After spheroidization the hexagonal lattice of niobium silicide Nb_5Si_3 is transformed into a tetragonal lattice (I4/m) with the parameter a = 0.6557 nm and c = 1.186 nm. The other phase components remain unchanged.

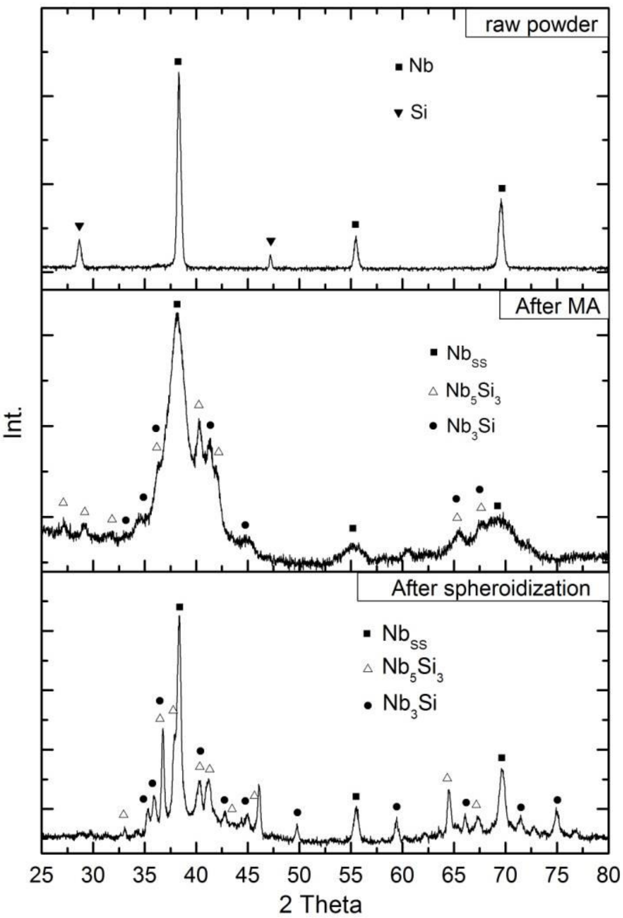

Figure-4. Changes in the phase composition of Nb-16Si alloy after mechanical alloying and spheroidization.

CONCLUSIONS

Experimental results show that plasma spheroidizing of Nb-16Si powders obtained by mechanical alloying is possible. The average size of the powders decreased from 60 to 45 μm after plasma treatment. XRD analysis has shown the presence of peaks of niobium solid solution (Nb_{SS}), as well as of niobium silicides Nb_5Si_3, Nb_3Si, which is confirmed by the results of studying the polished micro-sections.

Nb-16Si is initial alloy on which explored the possibility of preparing Nb-Si-alloy spherical particles by mechanical alloying and plasma spheroidization. Spherical Nb-Si powder alloy is a perspective material to manufacture products for the aerospace industry by additive technologies. Further research will be aimed at study the process of selective laser melting Nb-16Si spherical powder alloys and increasing the special characteristics of the Nb-16Si initial alloy by doping with Ti, Cr, Hf, Al, etc.

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REFERENCES

[1] Frazier W.E. 2014. Metal Additive Manufacturing: A Review. J. Mater. Eng. Perform. 23(6): 1917-1928.

[2] Wohlers T. Wohlers Report 2014. 3D Printing and Additive Manufacturing State of the Industry. Annual Worldwide Progress Report. Wohlers Associates, Inc.

[3] Uriondo A., Esperon-Miguez M., Perinpanayagam S. 2015. The present and future of additive manufacturing in the aerospace sector: A review of important aspects. Proc. Inst. Mech. Eng. Part G: J. Aerosp. Eng. 229(11): 2132-2147.

[4] Gao W. *et al*. 2015. The status, challenges, and future of additive manufacturing in engineering. Com. Aid. Des. 69: 65-89.

[5] Suryanarayana C. 2001. Mechanical alloying and milling. Prog. in Mat. Science. 46: 1-184.

[6] Chaturvedi V., Ananthapadmanabhan P. V., Chakravarthy Y., Bhandari S., Tiwari N., Pragatheeswaran A., Das A.K. 2014. Thermal plasma spheroidization of aluminum oxide and characterization of the spheroidized alumina powder. Cer. Int. 40(6): 8273-8279.

[7] Kumar S., Selvarajan V. 2008. Plasma spheroidization of iron powders in a non-transferred DC thermal plasma jet. Mater. Char. 59(6): 781-785.

[8] Yang S., Gwak J.-N., Lim T.-S., Kim Y.-J., Yun J.-Y. 2013. Preparation of spherical titanium powders from polygonal titanium hydride powders by radio frequency plasma treatment. Mater. Trans. 54(12): 2313-2316.

[9] Zhu H.L., Tong H.H., Yang F.Z., Wang Q., Cheng C.M. 2014. A comparative study on radio-frequency thermal plasmaspheroidization for two types of alumina ceramic powder. Adv. Mat. Res. 1058: 221-225.

[10] Pakseresht A. H., Rahimipour M. R., Vaezi M. R., Salehi M. 2016. Thermal plasma spheroidization and spray deposition of barium titanate powder and characterization of the plasma sprayable powder. Mat. Chem. Phys. 173: 395-403.