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THE DESIGN OF Ti-5AI-5M0-5V-3Cr ELEMENTAL POWDERS MIXTURE PROCESSING

The aim of the study was to indicate the influence of consolidation processes on microstructure and selected mechanical properties of powder metallurgy Ti-5Al-5Mo-5V-3Cr alloy, which was produced by blending of elemental powders method. Morphology of the mixture and its ingredients were examined using scanning electron microscopy. The consolidation of powders mixture was conducted using two approaches. The first consisted of the uniaxial hot pressing process, the second included two steps – uniaxial cold pressing process and sintering under argon protective atmosphere. Microstructural analysis was performed for both as-pressed compacts using light microscopy. Additionally, computed tomography studies were carried out, in order to examine the internal structure of compacts. Chosen mechanical properties, such as Vickers hardness and compression strength was also determined and compared. The conducted research proves that the proposed production method leads to obtain materials with no structural defects and relatively low porosity. Moreover, due to the proper selection of manufacturing parameters, favorable microstructures can be received, as well as mechanical properties, which are comparable to conventionally produced material with the corresponding chemical composition.

Keywords: titanium alloy; powder metallurgy; microstructure; mechanical properties; compaction

1. Introduction

Titanium and its alloys are extensively used in various fields of industry, due to their unique properties. Low density, high specific strength and excellent corrosion resistance allow applying titanium alloys as responsible elements of aircraft and vehicle construction as well as in the chemical industry. Moreover, titanium is considered as one of the most biocompatible materials, which makes it most often used metal for bone replacement and dental implants [1-3]. Nevertheless, the manufacturing costs of specific components from titanium alloys by conventional route is several times more expensive in comparison to aluminum alloys or stainless steel. Moreover, material lost, as the result of hot die forging and machining processes, can reach up to 90% [4]. The powder metallurgy (PM) methods are presently seen as promising in the production of titanium alloys components since they allow to manufacture near-net-shape parts with mechanical properties as good as those produced by a method based on ingot metallurgy. The Blended Elemental Powders Metallurgy (BEPM) approach results in minimizing technological steps and thus significantly decreasing manufacturing costs. Adopting the correct method of manufacturing of compacts as well as

an elaboration of appropriate pressing and sintering parameters provides to chemical and microstructural homogeneity. Good mechanical properties and decreasing residual porosity can be achieved by followed thermo-mechanical processing [5].

The Ti-5Al-5Mo-5V-3Cr (Ti-5553) alloy belongs to near β titanium alloys group. Good processability and hardenability caused that this material is widely applied in the aircraft industry, mostly in the landing gear assembly [6]. Moreover, the high content of β -stabilized elements, (vanadium, molybdenum, chromium) caused lower forging temperatures and loads. Previous studies reports, that flow behavior and further mechanical properties strongly depend on the initial microstructure of processed material [7-10]. The morphology of the α phase has the greatest influence on the behavior of the material during hot deformation. Zhao et al. [9] noticed that Ti60 alloy exhibits bigger stress value during hot compression, when the initial microstructure was composed of fine acicular α grains, as opposed to bimodal microstructure.

Taking the above under consideration, the selection of appropriate methods of production of initial material and control of its microstructure has a significant influence on further properties of titanium alloy components. Therefore, this work discusses

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the development of an effective approach in the production of Ti-5553 alloy by powder metallurgy methods. Two routes of manufacturing of compacts from elemental powders mixture were adopted. The analysis of microstructure and chosen mechanical properties of obtained semi-products were conducted.

2. Experimental methods

The initial material for the experimental research was Ti-5Al-5Mo-5V-3Cr (Ti-5553) alloy compacts made from the mixture of elemental powders of titanium, aluminum, molybdenum, vanadium and chromium, with nominal composition given in Table 1. For the manufacturing of the compacts, two different routes were used - hot pressing (HP) and cold pressing and sintering (CP and CP+S). The blending process was carried out for 90 minutes, with a rotational speed of 55 rpm. Additionally, the container with powder mixture contains tungsten carbide (WC) balls, in order to intensify the blending process. Two approaches of powders compaction were applied. The first of them relied on the uniaxial hot pressing process at the temperature of 1200°C under pressure of 25 MPa under the argon atmosphere for 45 min. The second approach contains processes of pressing and sintering. The uniaxial pressing process was conducted at room temperature, under various pressures. After compaction, the relative density of the obtained compacts was measured by the Archimedes method. Then selected compacts were sintered at the temperature of 1250°C for 4 h under a protective atmosphere of argon. The cooling of the compact after sintering was realized in two steps, inside of the furnace and on the air respectively.

In order to analyze the chemical composition of the Ti-5553 compact, the Energy-dispersive X-ray spectroscopy (EDS) analysis was performed, using a Hitachi HM-3000 scanning microscope. Computed tomography (CT) studies of the hot-pressed compact were carried out using v|tome|x L-450 tomograph, with a resolution of 50 µm. Ground and polished surface of the sample was etched using Kroll's reagent with the chemical composition of 2 vol% HF, 2 vol% HNO₃ and 94 vol% H₂O. Microstructural observations of as-pressed and sintered specimens were carried out on LEICA DM4000M light microscope. The average hardness of the compacted Ti-5553 powder mixture was determined by the Vickers method with a load of 19.6 kN. The strength of as-pressed and sintered materials was determined in the uniaxial compression tests, which were carried out at room temperature on the Zwick/Roell Z250 machine. Samples with dimensions 6 mm in height and 5 mm in diameter were machined from the investigated materials.

3. Results and discussion

3.1. Powder mixture characterization

The morphology of each ingredient and the powder mixture of the Ti-5553 alloy was shown on Fig. 1. Titanium powder (Fig. 1a) was produced by hydride-dehydride (HDH) technology and was an irregular shape with particle size no larger than 150 μ m. Smooth surface, atomized aluminum particles (Fig. 1b) had size up to 35 μ m, similar to molybdenum (Fig. 1c) in case of which particles had irregular shapes and rough surfaces. Greater size had vanadium (Fig. 1d) as well as chromium particles (Fig. 1e) and it was up to 150 μ m with irregular shape and irregular surface. As a result of the intensive powders blending process of the Ti-5553 alloy mixture (Fig. 1f), homogenous distributions of ingredients were obtained. Due to the application of WC balls, larger particles were broken, and smaller and softer, such as aluminum, was insert on the surface of the other mixture ingredients.

3.2. Influence of the method of consolidation on density and microstructure of compacts

In the result of the uniaxial HP process of Ti-5553 powders mixture, cylindrical compact with 77.5 mm in diameter and 41.5 high was produced. The measured relative density of HP compact was $98.2 \pm 0.4\%$ in reference to the theoretical density of solid material which was 4.68 g/cm^3 [15]. SEM observations (Fig. 2a) confirmed only a small amount of spherical residual porosity. Additionally, analysis of CT images did not reveal significant porosity or crack in a volume of compact (Fig. 2b)

The microstructure of as-pressed HP Ti-5553 alloy (Fig. 3a) consists of colonies of lamellar α grains (marked by arrow) located on β matrix and coarse α phase grains on the primary β grain boundaries (marked by a dotted line). Such microstructure was created as the result of slow air cooling after the hot pressing process. The α phase precipitations firstly appear on β grain boundaries and growth and block further growth of lamellar α grains formed inside primary β grain [11]. Nevertheless, microstructural observations, as well as EDS studies (Tab. 1), reveals inhomogeneity and regions which are rich in β -stabilizing elements, especially chromium.

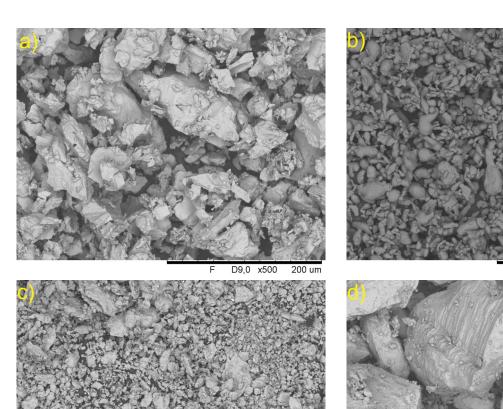
Taking the above under consideration HP material was subjected to a heat-treatment process that covered heating to the temperature of 1250°C, then compact was held at this temperature for 2 h under a protective atmosphere. Two types of cooling

TABLE 1

Nominal chemical composition of Ti-5553 alloy and average results from EDS study of compact (both in weight %)

	Al	Mo	V	Cr	0	Н	N	С	Ti
Nominal composition, %	4.4-5.7	4.0-5.5	4.0-5.5	2.5-3.5	<0.18	< 0.015	< 0.05	<0.1	bal.
EDS results, %	5.4	5.3	5.77	6.5	_	—		_	bal.





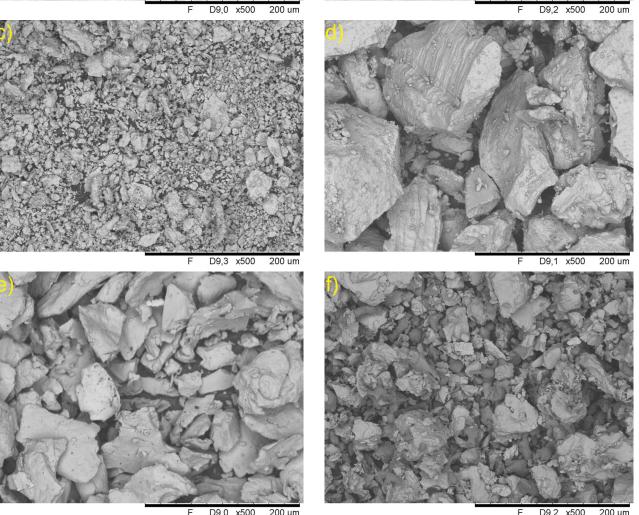


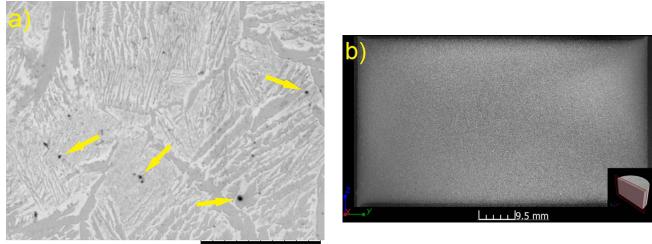
Fig. 1. Morphology of elemental powders: a) titanium, b) aluminium, c) molybdenum, d) vanadium, e) chromium, and f) powder mixture of Ti-5553 alloy

were applied: air cooling and water quenching. Microstructures of heat-treated Ti-5553 compact are presented on Fig. 3b and c. Such heat treatment was conducted in order to remodel the microstructure and homogenize chemical composition. The microstructure of heat-treated and water quenched Ti-5553 alloy compact (Fig. 3b) consisted of equiaxed β grains with thin plates of α phase on its boundaries. In the result of slow air cooling (Fig. 3c), β grains become coarse, and α plates on β grains boundaries transformed into thicker lamellas. Also, acicular α precipitations inside β grains appeared (marked by arrows).

A relatively high amount of α phase in the microstructure of Ti-5553 compact results from the oxygen contamination. This effect is a consequence of using a mixing process in the presence of balls with WC as a method of preparing a mixture of powders. Increased oxygen content may efficiently inhibit the β -stabilization effect of other alloying elements such as







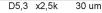


Fig. 2. SEM image with porosity indicated (a) and CT image (b) of hot pressed Ti-5553 alloy

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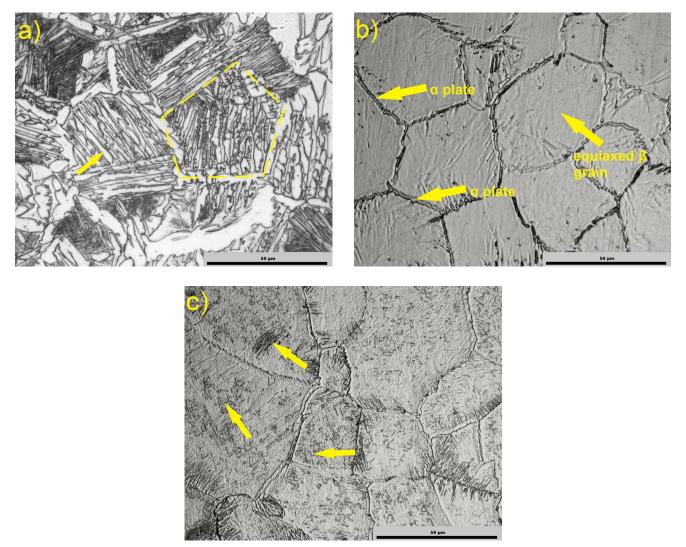


Fig. 3. Microstructures of as-pressed (a); heat-treated HP Ti-5553 alloy compact: b) water quenched, c) air-cooled

molybdenum, vanadium or chromium [12-14]. Nevertheless, applied heat treatment caused a significant reduction of amount of α phase and transformation of extant α grains in form of thin plates on β grains boundaries or acicular precipitations. Such

microstructure is considered as more beneficial in reference to further plastic deformation, than structure composed of lamellar α grains. Previous research [8,10] reported that the presence of lamellar microstructure increases the flow stress during deforma-

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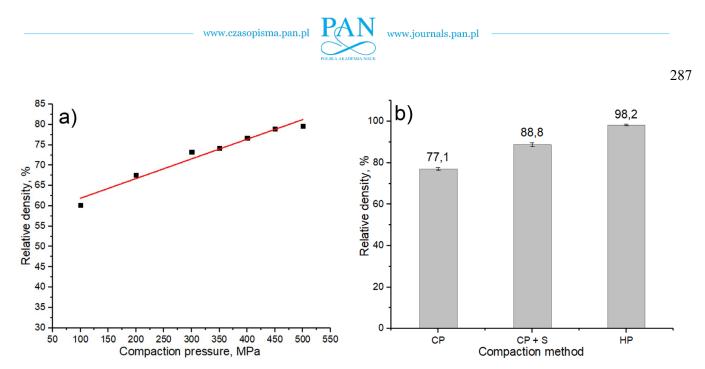


Fig. 4. a) Influence of cold compaction pressure on relative density of Ti-5553 alloy compacts, b) comparison of relative density depending on compaction method

tion due to the deformation mechanism of lamellar α grains, while structure consisting mainly of equiaxed grains causes a decrease of α/β interfaces, which leads to the reduction of flow stress.

In the result of the uniaxial CP process of Ti-5553 powder mixture, cuboid compacts with dimensions of 10 mm \times 10 mm \times 100 mm were manufactured. The influence of compaction pressure on pre-sintered compact was examined, the effects of compaction were shown on Fig. 4a. The relative density of the Ti-5553 compact growing almost linearly with increasing of compaction pressure to 450 MPa. Further increasing of pressure increase density insignificantly. Therefore, the pressure value of 450 MPa was chosen to made compacts for further research. The average relative density after the CP process at 450 MPa was 77.1 \pm 0.7%. After the sintering process (CP+S) relative density increased up to 88.8 \pm 0.9%. A comparison of relative density in relation to the compaction method was shown on Fig. 4b.

Microstructure observations of CP+S Ti-5553 alloy revealed a significantly higher amount of porosity in comparison

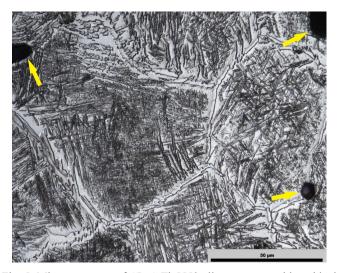


Fig. 5. Microstructures of CP+S Ti-5553 alloy compact with residual porosity indicated

to HP material, nevertheless the size of porosity does not exceed 50 μ m in diameter. No undissolved alloying particles were found. Microstructure (Fig. 5) was homogenous and mainly composed of acicular α precipitations on the β phase matrix. As in the case of HP material, new α grains firstly appear on primary β grains boundaries, then, in the result of slow furnace cooling precipitations grow inside of coarse β grain. In comparison to hot-pressed microstructure, the α grains are rather in form of needles, than lamellas. It was previously reported by Yang et al. [16], that in the case of β titanium alloys such microstructure exhibits better mechanical properties and higher elongation, than structure composed of only β grains.

3.3. Mechanical properties

Results of HV hardness measurements show significant differences between materials obtained by various methods (Fig. 6a). HP material exhibits the highest hardness value. Due to heat treatment and slow air cooling hardness insignificantly decrease. Water quenching after heat treatment does not affect the hardness of HP Ti-5553 alloy. In comparison to material obtained by the conventional ingot route, which assuming casting and metal forming, HP material exhibits similar values. CP+S material exhibits about 45% lower hardness in comparison to HP compact or conventional Ti-5553 alloy. Such a difference is mainly caused by significantly higher residual porosity of CP+S material.

Fig. 6b shows the stress-strain curves and compressive strength values of HP and CP+S Ti-5553 alloy, obtained at the ambient temperature. Both materials show a brittle fracture character, which allowed for the estimation of ultimate compressive strength. HP material exhibited greater strength value than CP+S compact (1894 MPa and 1537 MPa respectively). Despite high porosity, CP+S material exhibits relatively high true strain value and it was about 0.27. In comparison to the Ti-5553 alloy obtained by a conventional method, compressive yield strength for

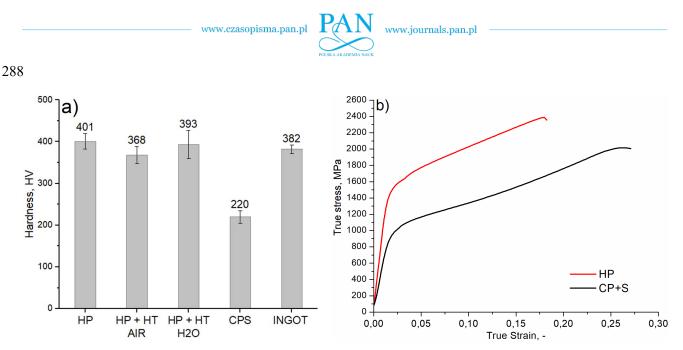


Fig. 6. Mechanical properties of compacts; a) hardness measurement results and b) obtained based on uniaxial compression test stress-strain curves and compressive strength values

HP material was similar (1138 MPa and 1243 MPa respectively). Slightly higher compressive yield strength value may be a result of increased chromium addition comparing to the reference alloy. Previous research [17-19] reported that chromium addition up to 10% may successfully increase mechanical properties in titanium alloys, which was mostly related to the solid-solution strengthening effect caused by chromium addition or the hardening effect of the ω phase formation resulting from cooling method. Compressive yield strength for CP+S compact was significantly lower and was comparable to commonly used dual-phase Ti-6Al-4V alloy (800 MPa and 897 MPa respectively) [2].

4. Conclusions

The influence of the consolidation approach of elemental powders mixture with a chemical composition corresponding to Ti-5Al-5Mo-5V-3Cr alloy was investigated. The use of two technological paths allowed the estimation of the impact of the compaction method on the relative density of the obtained materials, their microstructure and selected mechanical properties.

The main conclusions of this study can be summarized as follows:

- The production method based on the powder metallurgy technology, which assumed to use as starting materials of relatively cheap elemental powders and properly designed processes of mixing and hot pressing, leads to obtain material with high density as well as with no significant cracks or structural defects, while Ti-5Al-5Mo-5V-3Cr compact produced by cold pressing process and followed by sintering has about 10% higher value of porosity.
- 2) Microstructure observations reveal differences in the grain structure of each material. The microstructure of HP compact mainly composed of thick α plates, in contrast to acicular grains present in CP+S material. Also, it can be observed, that primary β grains in sintered Ti-5553 alloy are greater.

- 3) Comparison of the selected results of mechanical tests of the HP and CP+S compacts showed that the strength properties of both materials are relatively high, while the CP+S material showed higher plasticity. It is also worth to note, that HP Ti-5553 alloy has similar mechanical properties to conventionally produced material with corresponding chemical composition.
- 4) The conducted research allows to state, that application of powder metallurgy methods led to the production of material with satisfying mechanical properties and favorable microstructure. Proposed compaction technologies can be used for the production of a preform for further thermomechanical processing, during which the relative density can be increased and mechanical properties improved.

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REFERENCES

- Z.Z. Fang, J.D. Paramore, P. Sun, K.S.R. Chandran, Y. Zhang, Y. Xia, F. Cao, M. Koopman, Powder metallurgy of titanium – past, present, and future, Int. Mater. Rev. 63, 407-459 (2018).
- [2] R.R. Boyer, R.D. Briggs, The Use of β Titanium Alloys in the Aerospace Industry J. Mater. Eng. Perform. 22, 2916-2920 (2013).
- [3] M. Long, H. Rack, Titanium alloys in total joint replacement a materials science perspective, Biomaterials 19, 1621-1639 (1998).
- [4] J. Capus, Titanium powder developments for AM A round-up, Met. Powder Rep. 72, 384-388 (2017).
- [5] K. Zyguła, T. Śleboda, M. Wojtaszek, G. Korpała, Physical modeling of hot forging of cast and P/M Ti-6Al-4V alloy, METAL 2017–26th International Conference on Metallurgy and Materials, Conference Proceedings 1977-1982 (2017).
- [6] R.A. Antunes, C.A.F. Salvador, M.C.L. de Oliveira, Materials Selection of Optimized Titanium Alloys for Aircraft Applications, Mater. Res. 21 (2018).
- [7] S.S. Dheda and F.A. Mohamed, Effect of initial microstructure on the processing of titanium using equal channel angular pressing, Mater. Sci. Eng. A 528, 8179-8186 (2018).
- [8] P. Gao, M. Zhan, X. Fan, Z. Lei, Y. Cai, Hot deformation behavior and microstructure evolution of TA15 titanium alloy with nonuniform microstructure, Mater. Sci. Eng. A 689, 243-251 (2017).
- [9] Z.L. Zhao, H. Li, M.W. Fu, H.Z. Guo, Z.K. Yao, Effect of the initial microstructure on the deformation behavior of Ti60 titanium alloy at high temperature processing, J. Alloys Compd. 617, 525-533 (2014).
- [10] M. Jackson, N.G. Jones, D. Dye, R.J. Dashwood, Effect of initial microstructure on plastic flow behaviour during isothermal forging of Ti-10V-2Fe-3Al, Mater. Sci. Eng. A 501, 248-254 (2009).

- [11] F. Yang, B. Gabbitas, M. Dore, A. Ogereau, S. Raynova, L. Bolzoni, On microstructural evolution and mechanical properties of Ti-5Al-5V-5Mo-3Cr alloy synthesised from elemental powder mixtures, Mater. Chem. Phys. 211, 406-413. (2018).
- [12] T. Saito, H. Takamiya, T. Furuta, Thermomechanical properties of P/M β titanium metal matrix composite, Mater. Sci. Eng. A 243, 273-278 (1998).
- [13] R. Naseri, D.R.G. Mitchell, D.G. Savvakin, M.J.B. Nancarrow, T. Furuhara, A.A. Saleh, A.A. Gazder, E. V. Pereloma, The effect of β-phase condition on the tensile behaviour in a near-β Ti alloy produced by blended elemental powder metallurgy, Mater. Sci. Eng. A 747, 232-243 (2019).
- [14] E. Eisenbarth, D. Velten, M. Müller, R. Thull, J. Breme, Biocompatibility of β-stabilizing elements of titanium alloys, Biomaterials 25, 5705-5713 (2004).
- [15] Kaufman, J. Gilbert, (2012). Titanium Alloy Database. Knovel. Accessed April 1, 2019. Retrieved from https://app.knovel.com/ hotlink/toc/id:kpTAD00001/titanium-alloy-database/titaniumalloy-database.
- [16] F. Yang, S. Raynova, A. Singh, Q. Zhao, C. Romero, L. Bolzoni, Hybrid microwave sintering of blended elemental Ti alloys, Jom. 70, 632-637 (2018).
- [17] M. Hattori, S. Takemoto, M. Yoshinari, E. Kawada, Y. Oda, Effect of chromium content on mechanical properties of casting Ti-Cr alloys, Dent. Mater. J. 29, 570-574 (2010).
- [18] H.C. Hsu, S.C. Wu, S.K. Hsu, C.Y. Chen, W.F. Ho, Structure and mechanical properties of as-cast Ti-5Sn-xCr alloys, Mater. Sci. Eng. A 606, 157-164 (2014).
- [19] H. Hsu, S. Wu, S. Hsu, T. Lin, W. Ho, Structure and mechanical properties of as-cast Ti – 5Nb – x Cr alloys, Mater. Des. 51, 268-273 (2014).