UDK 669.14.018.8:669.112:620.17
Professional article/Strokovni članek

ISSN 1580-2949
MTAEC9, 46(5)519(2012)

EFFECT OF THE DELTA-FERRITE CONTENT ON THE TENSILE PROPERTIES IN NITRONIC 60 STEEL AT ROOM TEMPERATURE AND 750 °C

VPLIV VSEBNOSTI DELTA FERITA NA NATEZNE LASTNOSTI JEKLA NITRONIC 60 PRI SOBNI TEMPERATURI IN PRI 750 °C

Almaida Gigović-Gekić[1], Mirsada Oruč[2], Sulejman Muhamedagić[1]

[1]University of Zenica, Faculty of Metallurgy and Materials Science, Travnička cesta 1, Zenica, Bosnia and Herzegovina
[2]Metallurgical Institute "Kemal Kapetanović", Zenica, Bosnia and Herzegovina
almaida.gigovic@famm.unze.ba

Prejem rokopisa – received: 2011-10-19; sprejem za objavo – accepted for publication: 2012-03-15

This paper presents the results of the tensile testing of the austenitic stainless steel Nitronic 60 at room temperature and 750 °C in the solution-annealed condition. It also presents the results of the optical and SEM analyses of the tested samples. The microstructural analysis showed the presence of a delta-ferrite phase in the austenite matrix at room temperature. The content of the delta ferrite was calculated using a Feritscope MP30. The content of the delta ferrite depends on the chemical composition. An increase in the Si and Cr contents causes an increase in the delta-ferrite content. The results also show that an increase in the delta-ferrite content leads to an increase in the strength and a decrease in the ductility at room temperature. After testing the samples at 750 °C, the presence of a sigma phase was noticed. Precipitation of the sigma phase causes a slight increase in the strength and a decrease in the ductility of the tested material. An analysis of the fracture surface shows the presence of ductile fracture in the samples tested at room temperature and a combination of ductile and brittle fractures occurring at 750 °C.

Keywords: austenitic steel, Nitronic 60, tensile properties, SEM analysis, delta ferrite, sigma phase

Članek predstavlja rezultate nateznih preizkusov avstenitnega nerjavnega jekla Nitronic 60 v raztopno žarjenem stanju pri sobni temperaturi in pri 750 °C. Predstavljeni so tudi rezultati preiskav vzorcev s svetlobno in SEM analizo. Analiza mikrostrukture pri sobni temperaturi je pokazala prisotnost delta ferita v avstenitni osnovi. Vsebnost dela ferita je bila določena z uporabo naprave Feritscope MP30.Vsebnost delta ferita je odvisna od kemijske sestave. Povečanje vsebnosti Si in Cr poveča tudi vsebnost delta ferita. Iz rezultatov izhaja, da povečanje vsebnosti delta ferita poveča trdnost in zmanjša preoblikovalnost pri sobni temperaturi. Po preizkušanju pri 750 °C je bila opažena prisotnost sigma faze. Izločanje te faze rahlo poveča trdnost in poslabša preoblikovalnost preizkušenega materiala. Analiza prelomov kaže žilav prelom pri vzorcih, preizkušenih pri sobni temperaturi in kombinacijo žilavega in krhkega preloma pri 750 °C.

Ključne besede: avstenitno jeklo, Nitronic 60, natezne lastnosti, SEM-analiza, delta ferit, sigma faza

1 INTRODUCTION

Microstructure stability is the most important requirement needed to obtain proper mechanical properties for an austenitic stainless steel (ASS).[1] To achieve a stable microstructure, the samples are usually solution heat treated at a temperature between 1000 °C and 1120 °C [2] and then water quenched. The microstructure of Nitronic 60 is primarily monophasic, i.e., austenitic. However, precipitation of the delta ferrite (δ-ferrite) in an austenite matrix is possible, too. A higher volume fraction of the δ-ferrite can be achieved in the microstructure of the samples by changing the chemical composition of steel in terms of increasing the content of the δ-ferrite-stabilising elements[1] such as Cr, Si, Ti, Mo, etc. The presence of the δ-ferrite, which has a BCC crystalline structure, slows the grain growth and increases the strength properties of the steel because the interphase boundaries act as strong barriers to the dislocation motion[3]. During the annealing at 600–900 °C the intermetallic phases and carbides precipitate from the austenite and/or the δ-ferrite.[2,4] One of the most common phases in ASS is the sigma phase (σ-phase).[2,5] As a result of the heat-treatment temperature, the δ-ferrite can transform in the austenite and the σ-phase.[4–10] The σ-phase is an intermetallic compound with a complex, tetragonal, crystal structure. The chemical composition of this phase varies considerably and it is therefore difficult to define this phase in the form of unique formulas. At room temperature this phase is hard, brittle and nonmagnetic,[10] therefore having a negative effect on the mechanical properties especially on the toughness and ductility. The presence of the δ-ferrite reduces the incubation period of the precipitation of the σ-phase. The rate of the σ-phase precipitation from the δ-ferrite is about 100 times more rapid than the rate of the σ-phase precipitation directly from austenite[4]. The temperature interval, in which this phase occurs with most of the commercial steels, is between 590 °C and 870 °C, but the phase decomposes at temperatures above 1000 °C. To make the samples free of the σ-phase, the heat-treatment temperature has to be higher than 1000 °C, followed by rapid cooling.

The aim of the research is an investigation of the influence of δ-ferrite on the tensile properties of the tested materials at room and elevated temperatures because the Nitronic 60 parts are intended to operate at elevated temperatures.

For the research project we tested six melts with different contents of the alloying elements of Si, Cr, Mn and Ni in order to get the samples with different contents of the δ-ferrite.

2 EXPERIMENTAL WORK

The melts were prepared by using a vacuum induction furnace with an argon protective atmosphere with the chemical composition corresponding to the standard ASTM A276,[11] as shown in **Table 1**. The requirements relating to the chemical composition and the tensile properties of the tested material correspond to the requirements for the steel UNS S21800.[11] The average δ-ferrite content was determined with a Feritscope MP30 (Fisher, Germany) using broken specimens after the tensile testing at room temperature and 750 °C, as seen in **Table 1**. The content of the δ-ferrite was determined with the method that takes advantage of the fact that the δ-ferrite is magnetic, while austenite and the σ-phase are not. The average value was calculated on the basis of ten measurements.

A microstructural analysis was carried out with an Olympus optical microscope and a scanning electron microscope (Jeol JSM 5610 operating at 20kV, a take-off angle of 35°, and an elapsed livetime of 60 %) using broken specimens after the tensile testing. The samples for the microstructure analysis were prepared with the standard grinding and polishing techniques and etched with an aqua regia. In addition, a fractographic analysis was carried out on broken specimens after the tensile testing with the scanning electron microscope.

The tensile testing was performed on the samples obtained from a rod with a ϕ of 15 mm. Before the testing, the samples were solution annealed at 1020 °C for 60 min followed by water quenching. The samples were tested at room temperature and 750 °C. The tensile test was carried out on a universal hydraulic machine for static testing (200 kN). The sampling and testing procedures were realized in accordance with the standards BAS EN 10002-1/02 and BAS EN 10002-5/01.

3 RESULTS AND DISCUSSION

3.1 Microstructure

The microstructure analysis of the samples tested at room temperature shows the presence of a two-phase microstructure. The microstructure consists of δ-ferrite islands in an austenite matrix.[12] The δ-ferrite islands are elongated in the rolling direction. The precipitation of the δ-ferrite occurs mainly at the grain boundaries as shown in **Figure 1a**. **Figure 1b** shows the microstructure

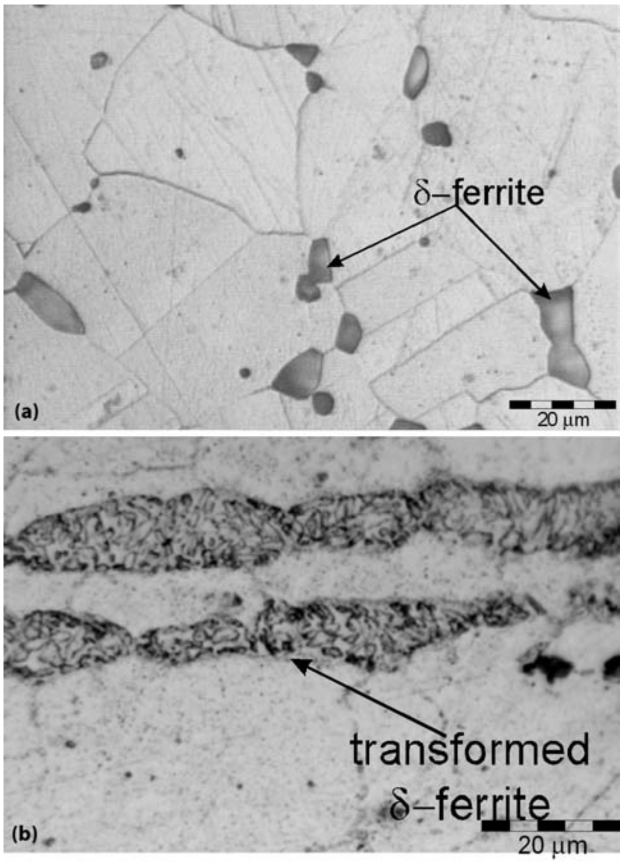

Figure 1: Microstructure of steel Nitronic 60 at: a) room temperature and b) 750 °C
Slika 1: Mikrostruktura jekla Nitronic 60 pri: a) sobni temperaturi in b) 750 °C

Table 1: Chemical composition and the average δ-ferrite content of steel Nitronic 60
Tabela 1: Kemijska sestava in povprečna vsebnost δ-ferita v jeklu Nitronic 60

Melt	Chemical composition, w/%								δ-ferrite /% (Room temp.)	δ-ferrite /% (750 °C)
	C	Si	Mn	Cr	Ni	P	S	N		
Prescribed ASTM A 276	max. 0.10	3.5–4.5	7.0–9.0	16.0–18.0	8.0–9.0	max. 0.06	max. 0.03	0.08–0.18		
1	0.04	4.41	7.4	18.0	8.1	0.007	0.005	0.18	10.43	0.52
2	0.04	3.74	8.6	18.0	8.0	0.007	0.005	0.16	6.30	0.31
3	0.04	4.25	8.4	16.0	8.8	0.006	0.010	0.14	3.37	0.77
4	0.05	3.5	7.9	16.9	8.6	0.005	0.005	0.12	1.30	0.58
5	0.04	3.5	7.2	16.6	8.0	0.006	0.012	0.15	1.14	0.59
6	0.05	3.8	8.9	17.0	9.0	0.007	0.005	0.16	0.82	0.46

of a sample tested at 750 °C exhibiting an austenite matrix and a transformed δ-ferrite phase.

The SEM analysis of the sample tested at 750 °C with a higher magnification is shown in **Figure 2**, confirming the transformation of the δ-ferrite. The nucleation of the sigma phases predominantly occurred at the austenite/δ-ferrite grain boundaries, because these grain boundaries and the interfaces are the high-energy regions. The average compositions of the constituent phases were determined with an EDS analysis and presented in **Table 2**. The results show that the σ-phase has the highest content of Cr and that the content of Ni is quite high, too. The austenite is rich in Ni and Mn, but depleted in Cr and Si. In the case of the δ-ferrite, it is depleted in Ni and rich in Cr.

Table 2: Chemical compositions of the constituent phases in mass fractions (w/%) obtained with an EDS analysis
Tabela 2: Kemijska sestava faz v masnih deležih (w/%), določena z EDS-analizo

Elements	Constituent phases		
	δ-ferrite	σ-phase	Austenite
Si	3.50	4.96	3.12
Cr	19.15	36.30	17.50
Mn	6.28	5.94	8.52
Ni	5.06	8.02	7.22

The δ-ferrite is decomposed into the σ-phase and the austenite with a eutectoid transformation.[9] The rate of the σ-phase precipitation from the δ-ferrite is more rapid than its precipitation directly from the austenite[4], and the δ-ferrite is not in the state of equilibrium at this temperature. The high susceptibility of the δ-ferrite phase to σ-phase formation is associated with the chemical composition of the δ-ferrite phase. This phase is rich in Cr and Si, which stimulate the formation of the σ-phase.[9,13] In the case of the alloys with the contents of Cr below 25 %, an addition of Mn and Ni can also stimulate the formation of the σ-phase.[4,14]

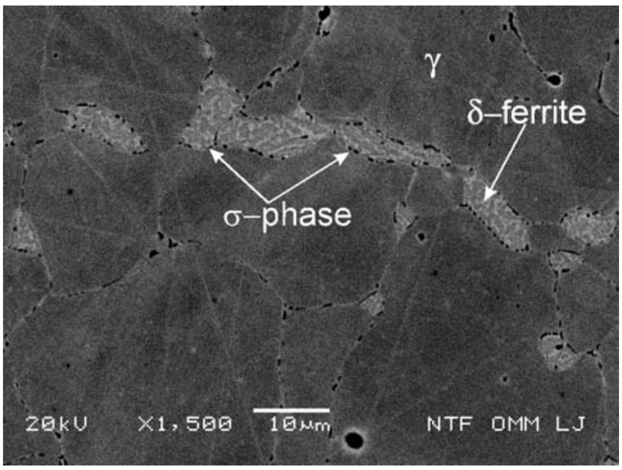

Figure 2: SEM microstructure samples tested at 750 °C
Slika 2: SEM-posnetek vzorca, preizkušenega pri 750 °C

3.2 Fracture surface

The analysis of the samples' fracture surfaces shows that the samples tested at room temperature have ductile fracture but the samples tested at 750 °C have intergranular brittle fracture as a consequence of the presence of the σ-phase with a small portion of a ductile fracture.[15] The ductile fracture is a result of the presence of austenite and the δ-ferrite, shown in **Figures 3a** and **3b**.

3.3 Mechanical properties

The results of the tensile testing at room temperature are in accordance with the standard ASTM A 276.[11] However, there is no data about the tensile testing at elevated temperature in the ASTM A 276 [11] standard. The results at room temperature indicate that an increase in the δ-ferrite content from 0.82 % to 10.43 % causes an increase in the tensile strength (TS) from 707 MPa to 821 MPa and an increase in the yield strength (YS) from 356 MPa to 467 MPa, while the reduction (Red.) and elongation (Elo.) are slightly reduced from 73 % to 63 % and from 55.3 % to 42.9 %, respectively, as shown in **Figure 4**.

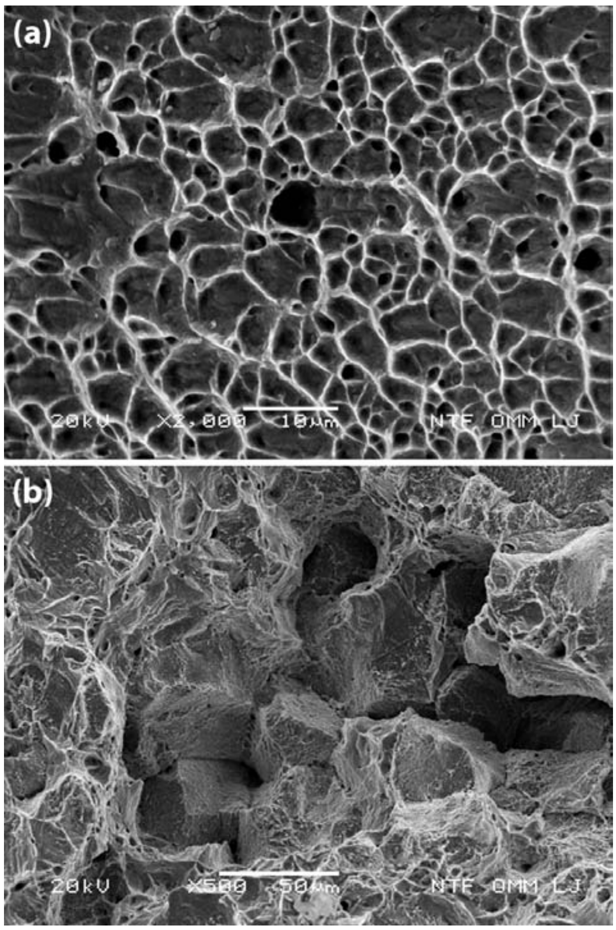

Figure 3: Fracture surface – SEM: a) room temperature and b) 750 °C
Slika 3: Površina preloma – SEM: a) sobna temperatura in b) 750 °C

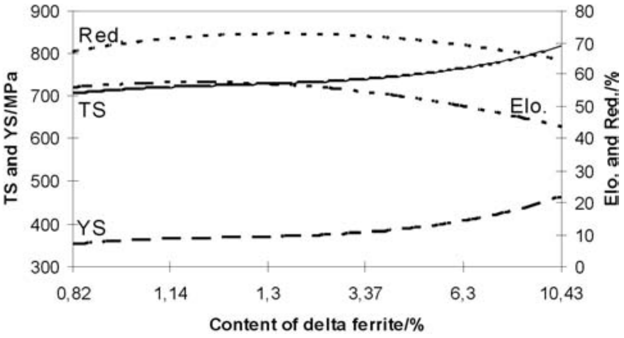

Figure 4: Effect of the δ-ferrite content on the tensile properties at room temperature
Slika 4: Učinek vsebnosti δ-ferita na natezne lastnosti pri sobni temperaturi

The highest value of the strength and the lowest value of ductile properties were found for the melt 1 with 10.43 % of the δ-ferrite. The values of the strength and ductility are almost constant being up to 3 % of the δ-ferrite. With an increase in the δ-ferrite content to over 3 %, the strength is increased and the ductility is decreased. The heating of the tested material at 750 °C causes a remarkable decrease in the strength and ductility of the samples in comparison with the test results obtained at room temperature. One of the reasons for this was the transformation of the δ-ferrite in the σ-phase. The analysis of the δ-ferrite content after the tensile testing at 750 °C shows that the σ-phase content increases with an increase in the δ-ferrite content. The content of the transformed δ-ferrite was about 60 % for the melts with the δ-ferrite content of up to 2 % and about 95 % for the melts with the δ-ferrite content of over 6 %. The average content of the transformed δ-ferrite for the melts from 2 % to 6 % was about 70 %. **Figure 5** shows that with an increase in the content of the transformed δ-ferrite from 77.15 % to 95.01 %, the tensile and yield strengths slightly increase from 245 MPa to 299MPa and from 180 MPa to 211 MPa, respectively. The yield strength is almost constant, being up to 77 %, while the tensile strength is decreasing. The ductility increases with an increase in the content of the transformed δ-ferrite from 43.9 % to 77.15 %. In the range between 77.15 % and 95.01 % the ductility slightly decreases. The value of the reduction is between 40 and 55 %, while the value of the elongation is between 25 and 45 %. The reason for the increase in the tensile strength is the precipitation of the σ-phase because the strength generally grows together with the growth of the precipitated intermetallic phases.[16,17] The intermetallic phases cause an increase in the strength by obstructing, or stopping, the movement of dislocations. It is known from the literature sources[13] that by controlling the distribution and morphology of the σ-phase, it is possible to improve the strength and ductility in the temperature range where the σ-phase precipitates.

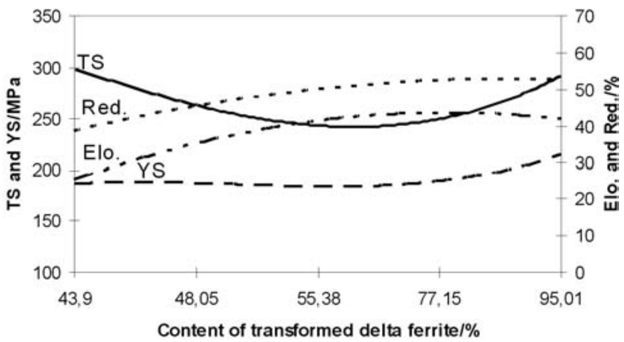

Figure 5: Effect of the content of transformed δ-ferrite on the tensile properties at 750 °C
Slika 5: Učinek vsebnosti pretvorjenega δ-ferita na natezne lastnosti pri 750 °C

Generally, the tensile properties of steel Nitronic 60 depend on the chemical composition of the alloy. By controlling the chemical composition of the alloy we control the δ-ferrite content in the steel. The content of the δ-ferrite increases with an increased concentration of the ferrite-stabilizing elements, such as Cr and Si, and with a decreased concentration of the austenite-stabilizing elements, such as Mn, Ni and N. The δ-ferrite content can become lower than 1 % if the contents of Ni and Mn are increased to the upper allowed limit and the contents of Cr and Si are decreased to the middle of the allowed limit,[18] as seen in **Table 1**. A reduction of the δ-ferrite content leads to a decrease in the strength and an increase in the ductility of steel at room temperature. At the elevated temperature (750 °C), the δ-ferrite transforms to the σ-phase, having a negative effect on the tensile properties because it is a hard and brittle phase.

4 CONCLUSION

In this study, the influence of the δ-ferrite on the tensile properties at room temperature and 750 °C was investigated. From the presented results it could be concluded that:

- The microstructure of the solution-annealed steel Nitronic 60 consisted of δ-ferrite islands in an austenite matrix at room temperature.
- An increase in the δ-ferrite content leads to an increase in the strength and a decrease in the ductility at room temperature, especially for the steel with over 3 % δ-ferrite.
- An increase in the content of Si and Cr causes an increase in the strength and a decrease in the ductility of steel due to the increased content of the δ-ferrite.
- The EDS analysis shows the presence of the σ-phase at the ferrite/austenite boundaries, which is a result of the δ-ferrite transformation at 750 °C. The percentage of the δ-ferrite decomposition in the samples was up to 90 %.
- The strength slightly increased and the ductility decreased after the content of the transformed δ-ferrite increased above 70 %.

- The SEM analysis also shows the presence of a mixture of the intergranular brittle fracture and ductile fracture at 750 °C, produced as a consequence of the presence of the sigma phase. The fracture surface of the samples tested at room temperature is ductile because of the presence of austenite and the δ-ferrite.

Acknowledgements

A part of the research described in this paper was conducted at the University of Ljubljana (Faculty of Natural Sciences and Engineering) as part of bilateral agreements between the Republic of Slovenia, and Bosnia and Herzegovina within the project "Application of new materials in the automotive industry" No: SLO-BA10-11-011.

5 REFERENCES

[1] J. Janovec, B. Šuštaršič, J. Medved, M. Jenko, Mater. Tehnol., 37 (**2003**) 6, 307–312

[2] R. L. Plaut, C. Herrera, D. M. Escriba, P. R. Rios, A. F. Padilha, Materials Research, 10 (**2007**) 4

[3] A. A. Astafev, L. I. Lepekhina, N. M. Batieva, https://springerlink.metapress.com/content/106486/?p=3803ac420c9f43dd80ca8a84319a1436&pi=0, https://springerlink.metapress.com/content/ g625036m8347/?p=3803ac420c9f43dd80ca8a84319a1436&pi=0

[4] A. F. Padilha, P. R. Rios, ISIJ International, 42 (**2002**) 4, 325–337

[5] H. S. Khatak, B. Raj, Corrosion of Austenitic Stainless Steels Mechanism, Mitigation and Monitoring, 2002

[6] F. Tehovnik, F. Vodopivec, L. Kosec, M. Godec, Mater. Tehnol., 40 (**2006**) 4, 129–137

[7] J. C. Tverberg, The Role of Alloying Elements on the Fabricability of Austenitic Stainless Steel, [cited:1. 4.2009.] Available from: www.csidesigns.com/tech/fabtech

[8] J. Pilhagen, A Literature Review of the Stainless Steel 21-6-9 and its Potential for Sandwich Nozzles, Master Thesis, Lulea, 2007

[9] F. Tehovnik, B. Arzenšek, B. Arh, D. Skobir, B. Pirnar, B. Žužek, Mater. Tehnol., 45 (**2011**) 4, 339–345

[10] S. Kožuh, M. Gojić, L. Kosec, RMZ-Materials and Geoenvironment, 54 (**2007**) 3, 331–344

[11] ASTM Standard A276-96, 1997 Annual Book of ASTM Standards, section 1. Iron and steel products, vol.01.03 Steel-Plate, Sheet, Strip, Wire, Stainless Steel Bar, ASTM, 1997

[12] A. Gigović-Gekić, M. Oruč, I. Vitez, Metallurgy, 50 (**2011**) 1, 21–24

[13] K. H. Lo, C. H. Shek, J. K. L. Lai, Materials Science and Engineering, R 65 (**2009**), 39–104

[14] Metals Handbook: Properties and Selection: Iron, Steels and High-Performance 10th. Edition, Alloys, vol.1, ASM American Society for Metals, 1990

[15] A. G. Gekić, M. Oruč, A. Nagode, H. Avdušinović, RMZ-Materials and Geoenvironment, 58 (**2011**) 2, 121–128

[16] M. Pohl, O. Storz, T. Glogowski, Materials Characterization, 58 (**2007**), 65–71

[17] S. C. Kim, Z. Zhang, Y. Furuya, C. Y. Kang, J. H. Sung, Q. Q. Ni, Y. Watanabe, I. S. Kim, Materials Transactions, 46 (**2005**) 7, 1656–1662

[18] M. Oruč, M. Rimac, O. Beganović, S. Muhamedagić, Mater. Tehnol., 45 (**2011**) 5, 483–487