

## THERMOMECHANICAL TREATMENT OF Ti-Nb-V-B MICRO-ALLOYED STEEL FORGINGS

### TERMOMEHANSKA OBDELAVA ODKOVKOV IZ MIKROLEGIRANEGA JEKLA Ti-Nb-V-B

**Marek Opiela**

Silesian University of Technology, Institute of Engineering Materials and Biomaterials, 18a Konarskiego Street, 44-100 Gliwice, Poland  
marek.opiela@polsl.pl

*Prejem rokopisa – received: 2013-09-30; sprejem za objavo – accepted for publication: 2013-10-09*

The work presents the research results of the influence of thermomechanical treatment through forging on the microstructure and mechanical properties of newly elaborated micro-alloyed steel containing 0.28 % C, 1.41 % Mn, 0.028 % Ti, 0.027 % Nb and 0.019 % V. The applied thermomechanical treatment makes it possible to obtain a fine-grained microstructure of austenite during the hot working and the production of forged parts that acquire an advantageous set of mechanical properties and a guaranteed crack resistance after controlled cooling from the finishing plastic-deformation temperature and the successive tempering. The forgings produced with the method of thermomechanical treatment, consecutively subjected to tempering in the temperature range from 550 °C to 650 °C, have  $YS_{0.2}$  from 994 MPa to 939 MPa,  $UTS$  from 1084 MPa to 993 MPa and  $KV^{40}$  from 77 J to 83 J.

Keywords: micro-alloyed steels, thermomechanical treatment, forged parts, mechanical properties

Delo predstavlja rezultate vpliva termomehanske obdelave s kovanjem na mikrostrukturo in mehanske lastnosti novo izdelanega mikrolegiranega jekla z 0,28 % C, 1,41 % Mn, 0,028 % Ti, 0,027 % Nb in 0,019 % V. Uporabljena termomehanska obdelava omogoča doseganje drobnostne mikrostrukture avstenita med vročo predelavo in izdelavo odkovkov, ki dobijo dobre mehanske lastnosti in zagotovljeno odpornost proti razpokam po kontroliranem ohlajanju iz končne temperature plastične deformacije in po popuščanju. Odkovki, izdelani po metodi termomehanske obdelave in nato popuščani v temperaturnem območju od 550 °C do 650 °C, imajo  $YS_{0.2}$  od 994 MPa do 939 MPa,  $UTS$  od 1084 MPa do 993 MPa in  $KV^{40}$  od 77 J do 83 J.

Ključne besede: mikrolegirana jekla, termomehanska obdelava, odkovki, mehanske lastnosti

## 1 INTRODUCTION

A lowering of the production cost is the basic reason for implementing economical technologies for the products made of constructional alloy steels with the methods of thermomechanical treatment. These methods consist of plastic working in the conditions adjusted to the steel chemical composition and micro-alloying with the consecutive direct cooling of the parts from the temperature of the plastic-deformation finish or after a particular time specified. This allows a reduction of the expensive heat treatment of products involving the tempering.<sup>1-4</sup>

The HSLA-type (high-strength low-alloy) micro-alloyed steels – containing up to 0.3 % C and 2 % Mn, micro-additions with a high chemical affinity to N and C, i.e., Nb, Ti and V in the amount of about 0.1 %, and sometimes also a slightly increased concentration of N and up to 0.005 % of B, increasing hardenability – are particularly useful for the production of forged parts with a fine-grained microstructure using the method of thermo-mechanical processing. The interaction of micro-additions in solid steel depends on their state influenced by the conditions of the performed plastic working. Micro-additions in the solid solution raise the temperature of the recrystallization of plastically deformed austenite. Moreover, the segregation of micro-additions at the grain boundaries decreases their mobility. Instead,

the micro-additions precipitating on dislocations in the form of dispersive particles of MX interstitial phases, slow down the course of dynamic recovery and possibly also dynamic recrystallization during the plastic deformation and additionally decrease the rate of thermal recovery and static or metadynamic recrystallization, limiting the grain growth of the recrystallized austenite in the intervals between the successive stages of deformation and after its completion. Dispersive particles of MX-type phases hamper the grain-boundary movement of the recrystallized austenite and, hence, it is possible to produce fine-grained-microstructure products additionally strengthened by carbonitrides.<sup>5-11</sup> The contribution of micro-additions to the precipitation strengthening can be calculated using the Ashby-Orowan relationship:

$$\sigma_p = \frac{0.538Gb_f^{1/2}}{\kappa} \ln\left(\frac{\kappa}{2b}\right) \quad (1)$$

where  $G$  is the shear modulus,  $b$  is the Burgers vector,  $f$  is the particle-volume fraction and  $\kappa$  is the mean diameter of particles.

When engineering the calculations, there is also another way of assessing the effect of the strengthening from the micro-alloyed particles:<sup>12</sup>

$$\sigma_p = K_p[M] \quad (2)$$

where  $[M]$  is the micro-addition content in the mass fraction and  $K_p$  is the constant.

Niobium is often used as a micro-addition in the steels subjected to various processes of thermomechanical treatment. Very dispersive Nb(C,N) precipitates have a very important role in this case. Therefore, the value of  $\sigma_p$  is often calculated with the assumption that the precipitation-strengthening effect depends mainly on this element. It should be noted that the effectiveness of Nb in the precipitation hardening is reduced by a Ti micro-addition forming both its carbides and nitrides as well as complex precipitates of (Ti,Nb)C and (Ti,Nb)(C,N).<sup>13</sup>

The state of niobium (in a solution or as a precipitate), determined by the reheating temperature, can affect the recrystallization, the grain growth and the  $\gamma \rightarrow \alpha$  transformation of austenite.<sup>14,15</sup> For example, the recrystallization and grain growth of austenite are significantly suppressed by the precipitation of NbC prior to the  $\gamma \rightarrow \alpha$  transformation. In addition, coarse NbC particles can be the preferred sites for the ferrite nucleation. In particular, the control of the austenite recovery and recrystallization is an important part of the grain-refinement technique in the modern thermo-mechanical controlling process.<sup>16</sup> An addition of Nb to steel is considered to have three primary effects:<sup>17,18</sup> (i) being an inhibitor of the austenite grain coarsening during reheating, (ii) suppressing the austenite recrystallization prior to the  $\gamma \rightarrow \alpha$  transformation through the strain-induced precipitation of NbC and (iii) causing the precipitation hardening from NbC in the low-temperature transformation step of the thermo-mechanical process. The strongest contribution to the strengthening is the refinement of the final microstructure (essentially ferrite grain size), which accounts for 80–90 % of  $\sigma_y$ . The key role of the precipitates is to provide the dispersion strengthening that is often generated in micro-alloyed steels by the NbC, VC, Nb(C,N) or V(C,N) particles, depending on whether N is added or not, with less than 20 nm in size.<sup>19</sup> Special attention also has to be paid to the vanadium micro-alloying addition. This element is easily added to liquid steel and its solubility during reheating is very high. The strengthening effect is enhanced as nitrogen is also added to the solution.

The final microstructure and mechanical properties strongly depend on the chemical composition, the controlled hot-working parameters and the cooling conditions of the steel. High strength, good ductility and good formability are developed in steel products during the manufacturing processes and, to achieve these goals, properly balanced quantities of micro-alloying additions and suitable thermomechanical processing schedules have to be carefully applied.<sup>20,21</sup>

## 2 EXPERIMENTAL PROCEDURE

The study was performed on newly elaborated micro-alloyed steel, intended for the production of forged

machine parts with a high strength, using thermomechanical treatment. The investigated steel contains 0.28 % C, 1.41 % Mn, 0.29 % Si, 0.008 % P, 0.004 % S, 0.26 % Cr, 0.11 % Ni, 0.22 % Mo, 0.025 % Al and Nb, Ti, V and B in the amounts of (0.027, 0.028, 0.019 and 0.003) %, respectively.

The investigated steel with the weight of 100 kg, molten in a VSG-100 type laboratory vacuum induction furnace and cast in an atmosphere of argon into quadratic hot-topped ingots with the following dimensions: top – 160 per bottom – 140 mm × 640 mm. The ingots were forged into 32 mm × 160 mm flat bars, with open-die forging in a high-speed hydraulic press, using a force of 300 MN.

The conditions of the hot processing and cooling, leading to the desired mechanical properties of the forgings, were selected with the following procedures:<sup>22</sup>

- analyses of the kinetics of the MX-type interstitial-phase precipitation in the solid state;
- an investigation of the process of hot working of steel with the method of continuous compression of the specimens at the rate of (1, 10 and 50) s<sup>-1</sup> in a temperature range from 1100 °C to 900 °C;
- an examination of the kinetics of strain hardening (recrystallization) and softening of plastically deformed austenite in the mentioned conditions,
- an investigation of the kinetics of the phase transformations of undercooled austenite.

The obtained results were used to develop two variants of forging with thermomechanical treatment of 160 mm × 32 mm flat bars into 14 mm thick flat bars, in the temperature range of 1100 °C to 900 °C at the strain rate of 3 s<sup>-1</sup>, applying 50 % of draft. The soaking temperature for forging was 1150 °C and the time was 45 min. In the first variant, segments of flat bars were hardened in water directly from the temperature of the forging finish, while, in the second variant, flat bars were isothermally held at the temperature of 900 °C for (10, 60 and 100) s after the forging finish and prior to hardening in water. Directly after quenching, the obtained flat-bar sections were tempered for 1 h at the temperatures of 550 °C and 650 °C.

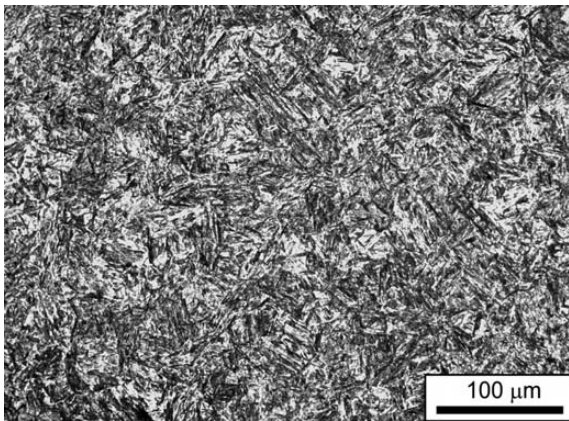
For the metallographic investigations of the specimens hardened after plastic deformation in the mentioned conditions and after high-temperature tempering, a Leica MEF 4A light microscope was used. The thin foils were examined with a TITAN80-300 FEI ultra-high resolution transmission electron microscope at the accelerating voltage of 300 kV.

Static tensile tests were performed with an INSTRON 1115 universal testing machine on the samples with a diameter of 8 mm and a gauge length of 40 mm. The impact testing at room temperature and at –40 °C was carried out on a Charpy pendulum machine with the initial energy of 300 J, using V-notch specimens with a cross-section of 8 mm × 10 mm. Brinell hardness was also determined for all the specimens.

### 3 RESULTS

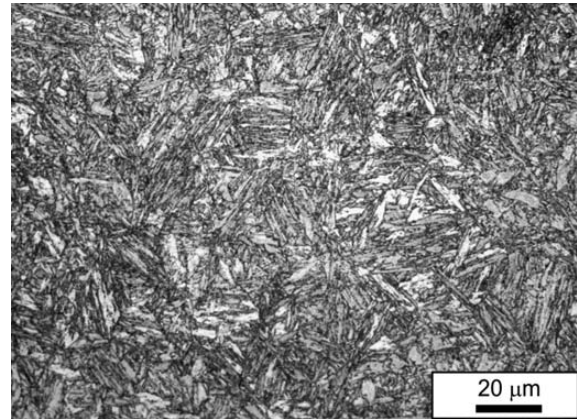
Metallographic examinations of the samples of Variant I of thermomechanical treatment, i.e., the ones quenched in water immediately after plastic deformation, revealed the microstructure of fine-lath martensite (**Figure 1**). The segments produced according to Variant I of thermomechanical treatment, subjected successively to tempering at the temperature of 650 °C, have the microstructure of the tempered martensite with the precipitates of carbide particles (**Figure 2**).

The analyses of the microstructure of thin foils revealed that the microstructure of steel, hardened directly from the forging temperature, consists of lath martensite and an uneven distribution of dislocation density in strongly plastically deformed austenite. Fine (Ti,Nb)C particles, located mainly at the boundaries of martensite laths (**Figure 3**) were observed in the martensite with a diversified spatial orientation of individual laths. Particles of similar chemical composition, morphology and size were also identified in the C-Mn-0.16Nb



**Figure 1:** Martensitic microstructure; Variant I of thermomechanical treatment: 900 °C, water

**Slika 1:** Martenzitna mikrostruktura; Varianta I termomehanske obdelave: 900 °C, voda



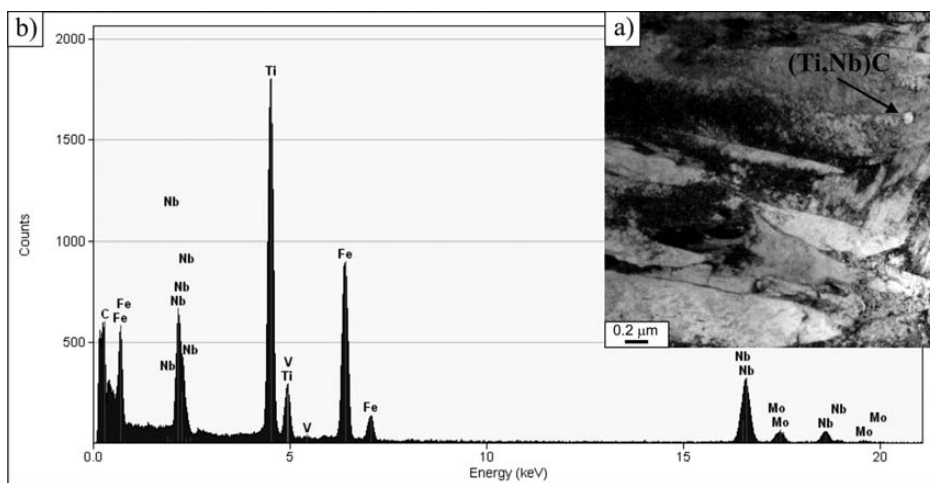
**Figure 2:** High-tempered martensite; Variant I of thermomechanical treatment: 900 °C, water; tempering temperature: 650 °C

**Slika 2:** Visoko popuščeni martenziti; Varianta I termomehanske obdelave: 900 °C, voda; temperatura popuščanja: 650 °C

steel.<sup>23</sup> Austenite in the form of thin films between the laths of martensite was observed. The Kurdjumov-Sachs crystallographic relationship between the retained austenite and martensite was confirmed.

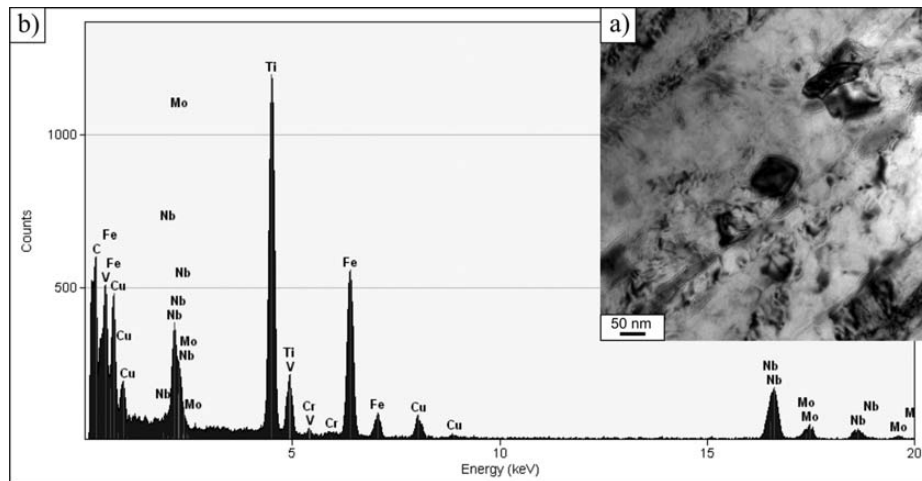
A similar morphology of lath martensite was found for the steel coming from the flat bar section obtained with Variant II of thermomechanical forging. Laths of different widths occur in the martensite packets. Some of the martensite laths show a fragmentation caused by high-angle subgrain boundaries formed into the statically recovered austenite that hampers their growth. As in the previous case, the (Ti,Nb)C and (Ti,Nb,V)C (**Figure 4**) particles with the size from 40 nm to 80 nm were found in the martensite.

The forgings produced in both variants of thermomechanical processing and the forgings quenched conventionally from the temperature of 900 °C have quite different microstructures after the tempering in the temperature range from 550 °C to 650 °C. The microstructure of the specimens taken from flat bar sections,



**Figure 3:** a) Carbide (Ti,Nb)C in the martensite, b) EDS spectrum; Variant I of thermomechanical treatment: 900 °C, water

**Slika 3:** a) Karbid (Ti,Nb)C v martenzitu, b) EDS-spekter; Varianta I termomehanske obdelave: 900 °C, voda



**Figure 4:** a) Carbide (Ti,Nb,V)C in the martensite, b) EDS spectrum; Variant II of thermomechanical treatment: 900 °C, 60 s, water  
**Slika 4:** a) Karbid (Ti,Nb,V)C v martenzitu, b) EDS-spekter; Varianta II termomehanske obdelave: 900 °C, 60 s, voda

**Table 1:** Results for the mechanical properties and impact-fracture energy of Charpy-V samples after thermomechanical forging and successive tempering

**Tabela 1:** Mehanske lastnosti in udarna žilavost Charpy-V vzorcev po termomehanskem kovanju in toplotni obdelavi

Variant	Treatment conditions			Tempering temperature °C	Mechanical properties				Impact energy	
	Charge heating/finish forging temperature, °C	Isothermal holding time, s	Cooling medium		$YS_{0.2}$ MPa	$UTS$ MPa	$TEI$ %	$RA$ %	$KV$ J	$KV^{-40}$ J
I	1150/900	–	water	550	973	1057	13.5	51.5	69.3	55.0
		–	water	650	909	976	14.0	52.0	81.7	68.6
II	1150/900	10	water	550	967	1063	13.6	49.6	91.6	71.3
		10	water	650	892	958	14.5	52.7	99.0	79.7
		60	water	550	994	1084	14.3	50.9	95.0	77.3
		60	water	650	911	974	15.1	50.2	108.7	82.6
		100	water	550	988	1077	13.0	49.6	95.7	75.7
		100	water	650	939	993	14.8	51.3	101.3	80.0

quenched directly after forging, after the tempering at the temperature of 550 °C, consists of the tempered martensite with a precipitation of granular and lamellar  $Fe_3C$  particles, distributed inside the grains and at the lath boundaries. The lamellar and granular precipitates, formed in such conditions, fulfill the matrix spatial dependences established by Bagariacki. An increase in the tempering temperature leads to a coagulation of cementite. The  $M_3C$  lamellar precipitates retain the privileged spatial orientation with ferrite, while the coagulated granular particles of this phase reveal random crystallographic orientation with respect to the matrix.

The samples obtained with the Variant-II forging of thermomechanical treatment have a similar microstructure in the tempered state. In this case, in the microstructure of the steel tempered at 550 °C there are thin lamellar precipitations of  $Fe_3C$  at the lath boundaries, while inside the laths of the recovered ferrite, dispersive lamellar particles of the phase with the privileged spatial orientation with the matrix are observed. Lamellar precipitates transform into granular  $Fe_3C$  particles at the lath boundaries and subgrain boundaries of the recovered

ferrite with an increase in the tempering temperature. Moreover, the precipitates of (Ti,Nb)C with a morphology similar to that after thermomechanical processing were found after tempering.

The steel microstructure in both hardened and tempered state significantly affects the mechanical properties of the flat bar sections obtained with both thermomechanical treatments. The results of the investigation of the mechanical properties and the impact energy of the Charpy-V samples, taken from the forgings are listed in **Table 1**. The data presented in this table show that the flat bar sections quenched in water directly from the temperature of the forging finish (Variant I of thermomechanical treatment), have the following properties after the tempering at 550 °C:  $YS_{0.2}$  of about 973 MPa,  $UTS$  of about 1057 MPa,  $TEI$  of about 13.5 % and  $RA$  of about 51.5 %. The flat bar sections obtained from Variant II of thermomechanical processing have higher mechanical properties and a distinctly higher crack resistance in the tempered state. However, the best set of mechanical properties and crack resistance was found for the forging isothermally held at the temperature of

900 °C for 60 s prior to hardening in water when, after the tempering in the temperature range from 550 °C to 650 °C, the following properties were obtained:  $YS_{0.2}$  from 994 MPa to 911 MPa,  $UTS$  from 1084 MPa to 974 MPa,  $KV$  from 95 J to 109 J and  $KV^{-40}$  from 77 J to 83 J.

The examination of the influence of the applied processing variant and the tempering temperature on the hardness show that the highest hardness – of approximately 330 HBW – is obtained for the forgings of the Variant-II thermomechanical treatment, including isothermal holding at the forging-finish temperature for 60 s prior to water quenching, and tempering at 550 °C. An increase in the tempering temperature for a forging up to 650 °C results in a mild decrease in the hardness, to about 300 HBW.

#### 4 CONCLUSIONS

The implemented thermomechanical processing allows a production of forged products with the following properties achieved after controlled cooling from the temperature of the plastic deformation finish and a subsequent tempering in the temperature range from 550 °C to 650 °C:  $YS_{0.2}$  from 994 MPa to 939 MPa,  $UTS$  from 1084 MPa to 993 MPa,  $TEI$  from 14.3 % to 15.1 %,  $RA$  from 51.5 % to 52.7 %,  $KV$  from 96 J to 109 J and  $KV^{-40}$  from 77 J to 83 J. The high strength in such a state at a high crack resistance, also at a decreased temperature, is noteworthy.

The best set of mechanical properties and crack resistance was obtained for the forging isothermally held at 900 °C for 60 s prior to quenching in water, and subsequently subjected to a tempering at 650 °C.

The relatively low hardness of the steel after a high-temperature tempering should not cause difficulties during the machining of forgings.

Microstructural observations of thin foils using transmission electron microscopy revealed that the microstructure of the steel in the quenched state consisted of lath martensite with a high density of dislocations and a considerable amount of precipitates. In the examined steel, the particles of (Ti,Nb)C and (Ti,Nb,V)C revealed precipitation on dislocations during plastic deformation, decreasing the rate of dynamic recovery, possibly dynamic recrystallization and, after hot working, also decreasing the rate of recovery and static or metadynamic recrystallization. The identified particles with the

sizes between 40 nm and 80 nm significantly increase the precipitation strengthening; however, they limit the grain growth of the recrystallized austenite and stimulate a formation of fine-grained microstructures.

The investigation makes it possible to develop an industrial technology for the forgings with high mechanical properties and a guaranteed crack resistance, also at a decreased temperature, using thermomechanical treatment.

#### 5 REFERENCES

- <sup>1</sup> A. Ghosh, S. Das, S. Chatterjee, B. Mishra, P. Ramachandra Rao, *Mater. Sci. Eng. A*, 348 (2003), 299–308
- <sup>2</sup> J. Adamczyk, E. Kalinowska-Ozgowicz, W. Ozgowicz, R. Wusatowski, *J. Mater. Proc. Tech.*, 54 (1995), 23–32
- <sup>3</sup> M. Cabibbo, A. Fabrizi, M. Merlin, *J. Mater. Sci. Tech.*, 43 (2008), 6857–6865
- <sup>4</sup> P. Skubisz, H. Adrian, J. Sińczak, *Arch. Metall. Mater.*, 56 (2011), 93–107
- <sup>5</sup> T. Gladman, *The physical metallurgy of microalloyed steels*, 1<sup>st</sup> ed., The University Press, Cambridge 1997
- <sup>6</sup> A. Grajcar, W. Borek, *Arch. Civ. Mech. Eng.*, 8 (2008), 29–38
- <sup>7</sup> B. Eghbali, A. Abdullah - Zadeh, *J. Mater. Proc. Tech.*, 180 (2006), 44–48
- <sup>8</sup> P. S. Bandyopadhyay, S. K. Ghosh, S. Kundu, S. Chatterjee, *Metall. Mater. Trans. A*, 42 (2011), 2742–2752
- <sup>9</sup> D. A. Skobir, M. Godec, M. Balcar, M. Jenko, *Mater. Tehnol.*, 44 (2010) 6, 343–347
- <sup>10</sup> D. K. Matlock, G. Krauss, J. G. Speer, *J. Mater. Proc. Tech.*, 117 (2001), 324–328
- <sup>11</sup> A. Grajcar, S. Lesz, *Mater. Sci. Forum*, 706–709 (2012), 2124–2129
- <sup>12</sup> P. D. Hodgson, M. R. Barnett, *ISIJ Int.*, 32 (1992), 1329–1336
- <sup>13</sup> H. Kejian, T. N. Baker, *Mater. Sci. Eng. A*, 169 (1993), 53–58
- <sup>14</sup> S. C. Hong, S. H. Lim, K. J. Lee, D. H. Shin, *Mater. Sci. Eng. A*, 355 (2003), 241–248
- <sup>15</sup> O. Kwon, A. J. DeArdo, *Acta Metall. Mater.*, 39 (1991), 529–538
- <sup>16</sup> M. Maruyama, R. Uemori, M. Sugiyama, *Mater. Sci. Eng. A*, 250 (1998), 2–7
- <sup>17</sup> S. Takaki, K. Kawasaki, Y. Kimura, *J. Mater. Proc. Tech.*, 117 (2001), 359–363
- <sup>18</sup> W. M. Rainforth, M. P. Black, R. L. Higginson, E. J. Palmiere, C. M. Sellars, *Acta Mater.*, 50 (2002), 735–747
- <sup>19</sup> M. MacKenzie, A. J. Craven, C. J. Collins, *Scri. Mater.*, 54 (2006), 1–5
- <sup>20</sup> R. Kuziak, T. Bołd, Y. Cheng, *J. Mater. Proc. Tech.*, 53 (1995), 255–262
- <sup>21</sup> J. Adamczyk, *Journal of Achievements in Materials and Manufacturing Engineering*, 14 (2006), 9–20
- <sup>22</sup> M. Opiela, A. Grajcar, *Arch. Civ. Mech. Eng.*, 12 (2012), 327–333
- <sup>23</sup> S. Vervynckt, K. Verbeken, P. Thibaux, *Mater. Sci. Eng. A*, 528 (2011), 5519–5528