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Performance of a Matrix Type High Speed Steel after Deep Cryogenic and Low Tempering Temperature

Kaweewat Worasaen[1,a], Andreas Stark[2,b], Karuna Tuchinda[1,c] and Piyada Suwanpinij[1,d*]

[1]The Sirindhorn International Thai-German Graduate School of Engineering (TGGS), King Mongkut's University of Technology North Bangkok, 1518 Pracharaj 1, 10800, Bangkok, Thailand

[2]Helmholtz-Zentrum Geesthacht Zentrum für Material- und Küstenforschung GmbH, Max-Planck-Straße 1, 21502 Geesthacht, Germany

[a]kaweewat.w-mpe2016@tggs.kmutnb.ac.th, [b]andreas.stark@hzg.de, [c]karuna.t@tggs.kmutnb.ac.th, [d]piyada.s.mme@tggs-bangkok.org

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Abstract. A matrix type high speed steel YXR3 designed for a combination of wear resistance and toughness is investigated for its mechanical properties after hardening by deep cryogenic treatment follow by tempering. The deep cryogenic quenching carried out at -200 °C for 36 hours and the single step tempering results in an obvious improvement in wear resistance while balancing the toughness, comparing with the conventional quenching followed by a double tempering treatment. The quantitative image analysis reveals little difference in the MC carbide size distribution between tempering at different temperatures. The synchrotron high energy XRD confirms the MC type carbide with some evolution in its orientation together with tempered martensite approaching the BCC structure at higher temperatures. In contrary to the conventional quenching and tempering, the lowest tempering temperature at 200 °C yields a moderate drop in hardness with increase in surface toughness proportionally while exhibiting exceptional wear resistance. Such thermal cycle can be recommended for the industry both for the practicality and improved tool life.

Introduction

A matrix type High Speed Steel (HSS) such as the Hitachi's YXR3 grade has been designed for the exceptional toughness that no breakage takes place before significant wear in case of forging tools [1]. Its hardness and toughness lie between other tool steels such as the AISI M2, T1, H10 and D2, etc. It contains significantly lower amount of alloying elements than other conventional groups as well as significantly lower amount of the MC carbide. The conventional heat treatment practice has been performed by quenching and double tempering in order to eliminate the retained austenite and afterwards tempered the newly formed martensite after the former tempering step. High temperature tempering is usually necessary for wear resistance improvement, usually at 560-600 °C [2].

Subzero quenching and shallow cryogenic treatment (SCT) have been introduced since 1937 [3] in order to avoid the retained austenite and investigated the improved properties after the tempering in different tool steels. The term Deep Cryogenic Treatment (DCT) in this paper refers to the very low temperature treatment, i.e., - 200 °C while the term SCT means the treatment at higher temperature around -70 °C. The deeper temperature allows more retained austenite to transform into martensite, in case of lower M_f temperature. Moreover, both finer and more uniform martensite laths carbide particles have been also reported, such as that in [4] that investigated H13 hot work tool steel. Its superior wear resistance improvement than that by SCT is in accordance to that found in D6 tool [5] and T1 HSS [6]. The findings in the uniform finely distributed of secondary carbides are in accordance to many other works including [7, 8] on other tool steel groups. The improvement in hardness is known to be due to uniformity rather than higher level [9]. The internal stress in martensite is believed to be lowered [10]. The key point that at the temperature near 0 K, crystals become nearly perfect [11] can be the mechanism that differentiates the DCT from SCT. The incomplete

understanding of the phenomena at cryogenic temperatures is a hindrance for its market acceptance although it is an inexpensive process [12] bringing about appearant materials performance. This paper therefore analyzes the evolution of wear resistance versus the hardness and toughness of the matrix type HSS YXR3 after DCT and tempering beside the investigation of its microstructure under the research questions from the industry.

Methodology

The commercial grade of matrix type HSS, YXR3 by Hitachi, was investigated for its performance after DCT and tempering. It contains the following chemical composition in mass-%: 0.62C, 1.36Si, 0.41Mn, 4.15Cr, 2.55Mo and 1.69V as reported by optical emission spectroscopy. The samples were available in thin cylindrical shape with 20 mm in diameter and 5 mm in thickness after conventional quenching and tempering processes. The studied thermal cycles listed in Table 1 were treated in a vacuum furnace for the austenitizing and quenching to ambient temperature within 60 minutes. The DCT process was treated afterwards by gradual cooling to -200 °C with helium gas. The tempering was carried out in a resistance furnace after 36 hours at the DCT temperature.

Table 1 The thermal cycles of the investigated samples

Sample	Austenitizing	Conventional Quenching	DCT	Tempering
DCT	1140 °C for 40 min	To ambient within 60 min	- 200 °C for 36 hrs	No tempering
T200				200 C for 2 hrs
T560				560 C for 2 hrs
T600				600 C for 2 hrs

The mechanical properties of the tempered samples after the DCT were analyzed compared with the samples from the conventional quenching and tempering (austenitized at 1140 °C, quenched to room temperature, first tempering at 540 °C for 4 hours, second tempering at 520 °C for 4 hrs.) The new cycles studied in this paper are listed in **Table 1**. Each tempered sample was then measured for the micro Vickers hardness (HV1) with a dwell time of 15 s at 5 positions. The wear resistance was evaluated by a ball-on-disk test with a 6 mm tungsten carbide ball applied a load of 5 N to measure the wear loss in volume. A scratch test was employed to calculate the surface toughness according to Akono [13-15] and our previous work [16]. The lattice parameter identification of the matrix and the MC carbide was investigated by means of the synchrotron High Energy X-ray Diffraction (HEXRD) with an energy of 100 keV at P07 beamline at PETRAIII ring situated at DESY. It is equivalent to a monochromatic X-ray with a wavelength of 0.124 A. The X-ray beam with a size of 1×1 mm diffracted through the sample thickness of 5 mm. The image analysis for the MC carbide size distribution was performed manually using Digimizer software on the micrographs obtained by Scanning Electron Microscopy (SEM) in backscattered mode at the magnification of 5,000x for large sampling area. The micrographs were taken in the FEI Quanta 450 equipped with LaB_6 emitter on the sample surface polished until 1 μm and etched with Villela acid (1 g picric acid, 5 ml hydrochloric acid, 100 ml ethanol).

Results

The hardness versus surface toughness are represented in Fig. 1. It can be seen that the hardness drops from 840 to 770 HV1, namely, around 10 percent with the increased tempering temperatures from none to 600 °C for 2 hours and drops to 820 HV1 after the tempering at 200 °C. The surface toughness increases inversely to the change in hardness due to the softening effect. In contrast, the wear rate in the sample tempered at 600 °C amounts to double of that at 200 °C. This emphasizes that the low temperature tempering remains significantly the wear resistance in addition to the restoration of the toughness of the YXR3 steel apart from the clear evidence that the DCT upgrades the wear resistance as a whole. The large standard deviation in the wear rate in the as-received sample after

the conventional quenching and double tempering can be attributed to the inhomogeneity in the microstructure and texture revealed by HEXRD.

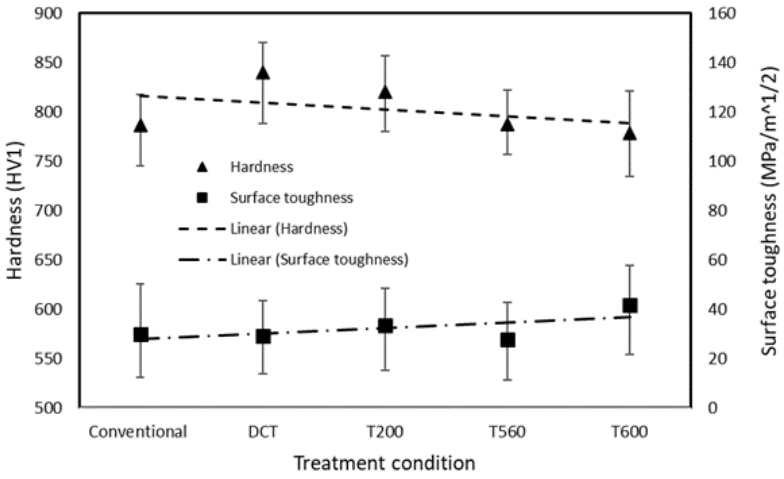

Fig. 1 The hardness and surface toughness observed in samples tempered at different temperatures

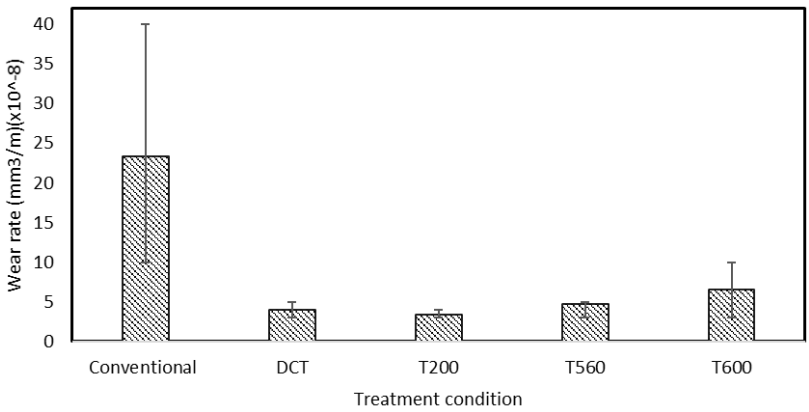

Fig. 2 The wear rate in the samples after DCT and tempering at different temperatures compared with that from the as-received condition (after conventional quenching and double tempering)

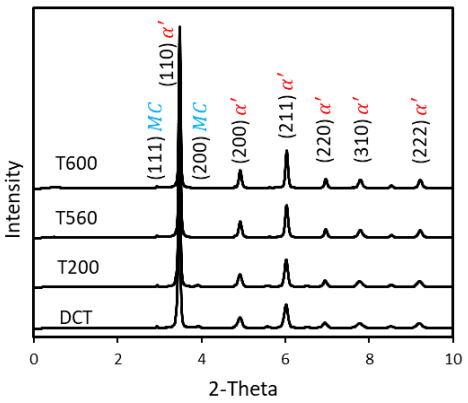

Fig. 3 X-ray diffraction patterns for DCT and tempered specimens

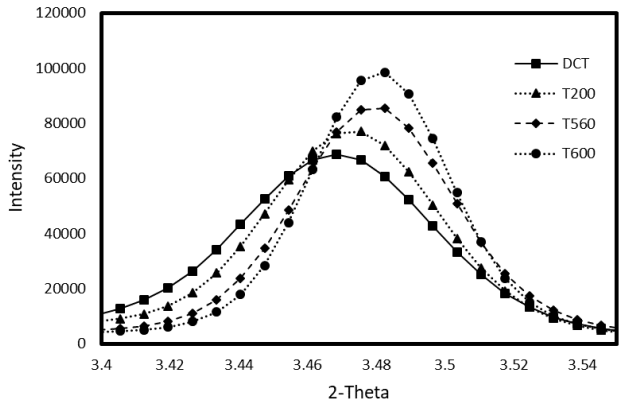

Fig. 4 The sharpening phenomenon of the (110) peak of α'martensite becomes more obvious at higher tempering temperatures due to the decrease in the tetragonality of its BCT lattice

The 1-D diffraction pattern obtained from the HEXRD is depicted in Fig. 3. It shows the major (110) peak of the tempered martensite as well as the co-existence of the MC carbide at (111) and (200). Please note that this minor phase cannot be detected in any other X-ray diffractometer. A close-up of the (110) peak sharpening of the tempered martensite at higher temperatures is revealed in

Fig. 4. This confirms that the inhomogeneity in the martensite gradually decreases and the tetragonality of the BCT structure distorted by the high carbon content is reduced that the lattice of tempered martensite approaches that of BCC [17]. This visualizes the evolution in martensite lattice after 2-hour tempering apart from the well-known criterion that tempering martensite at higher temperature than 350 °C yields BCC structure [18]. The close up on the peaks of the MC carbide at (111) and (200) in Fig.5 showing the decline in intensity at (200) and increases at (111) can be rationalized for the rearrangement in its structure during the tempering and hence the amount of the MC carbide shall not be affected. This point corresponds to the finding from the image analysis analyzed from the SEM results for more information on the size distribution of the MC carbide. The histogram of the size distribution plotted in Fig. 7 shows very fine size of mostly less than 1 μm at all tempering temperatures infers that its size distribution and amount are barely affected by the tempering.

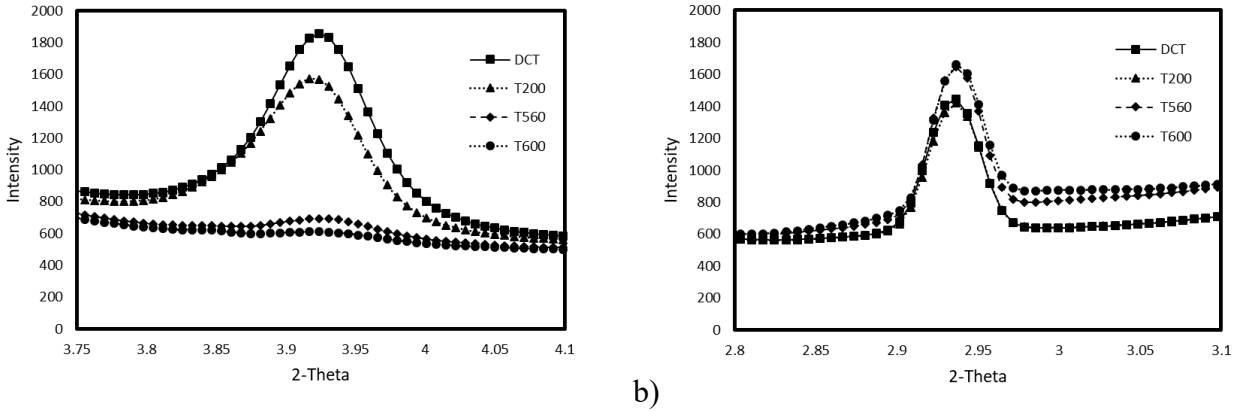

Fig. 5 a) The inclination of the (200) peak of the MC carbide phase and b) the sharpening of the (111) peak of the same phase

The substantial increase in wear resistance after tempering at the lowest temperature, therefore, possibly relates to other undetectable carbide formation as discussed in the literature. Meng reported a sharp improvement in wear resistance due to eta carbide at a similar tempering temperature as investigated in [19] after a cryogenic treatment. The eta orthorhombic carbide is well-known to form during the tempering of martensite in the lowest temperature level, up to 260 °C [20] and has been mentioned in several other research works [21-23]. The acceleration of the eta carbide formation can be contributed from the lattice compression at the cryogenic temperature that promotes the clustering of carbon atoms that behave as nuclei for further precipitation during the tempering. It has to be noted that the micro hardness measurement can mainly reflects the response from the matrix.

Conclusions

The innovative DCT followed by a low temperature tempering at 200 °C results in a great improvement in wear resistance in the YXR3 matrix type HSS while restoring the toughness due to the combination of softening in martensite, possible submicro carbide precipitation such as the well-known eta phase. This offers superior wear property to the conventional quenching and tempering that usually higher tempering temperature have been selected in addition to the short tempering cycle. The decrease in hardness and the correlated improvement in toughness is caused by the transformation of BCT into near BCC lattice as revealed clearly by the HEXRD. The MC carbide is not influenced by the tempering in terms of amount and size but the decline in a diffraction peak and increase in the other peak of the MC carbide micro precipitate should give a hint for its preferred orientation. The proposed heat treatment cycle can be recommended to the industry for the improvement in tool life as well as the production time saving.

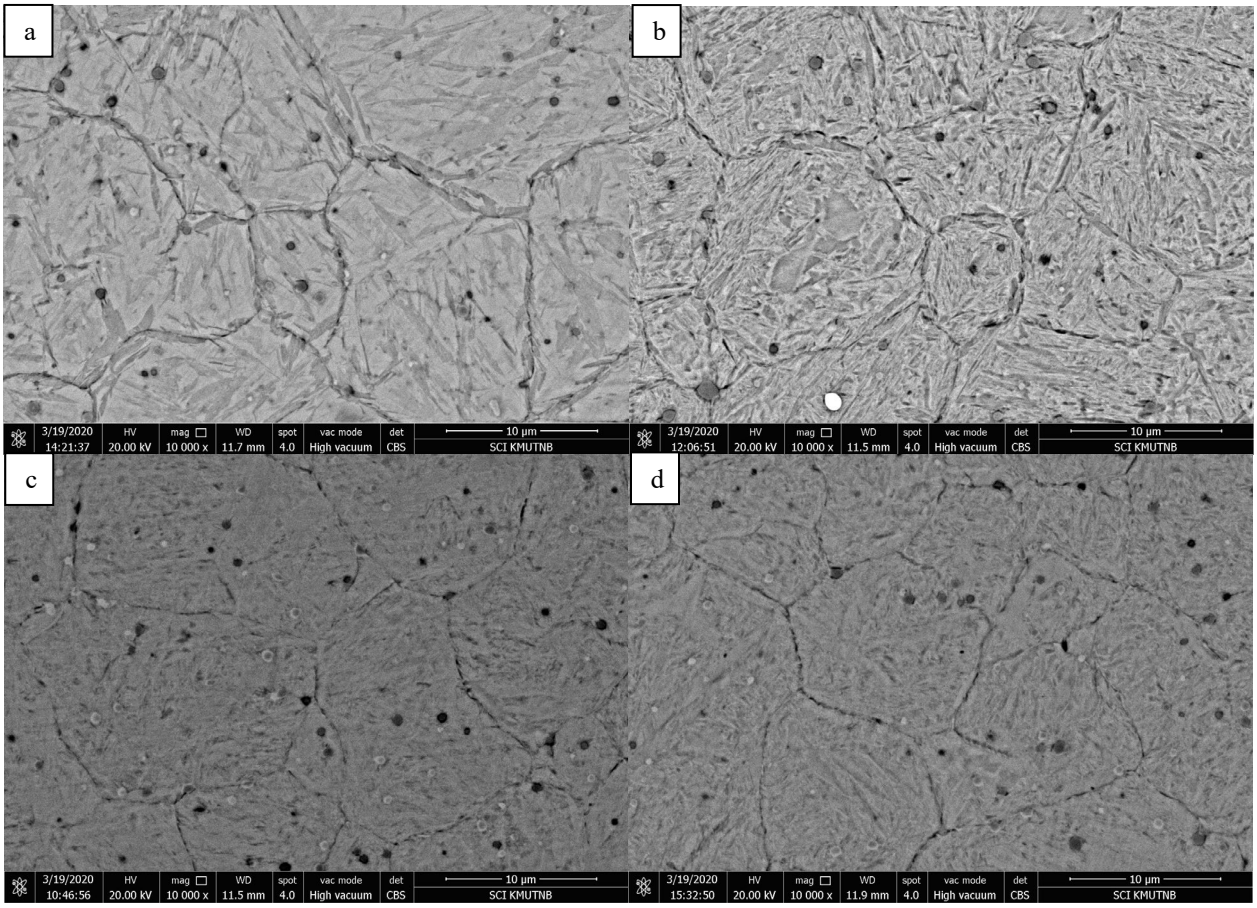

Fig. 6 SEM images at 10000x of the samples after (a) no tempering (DCT), (b) tempering at 200 °C, (c) 560 °C and (d) 600 °C

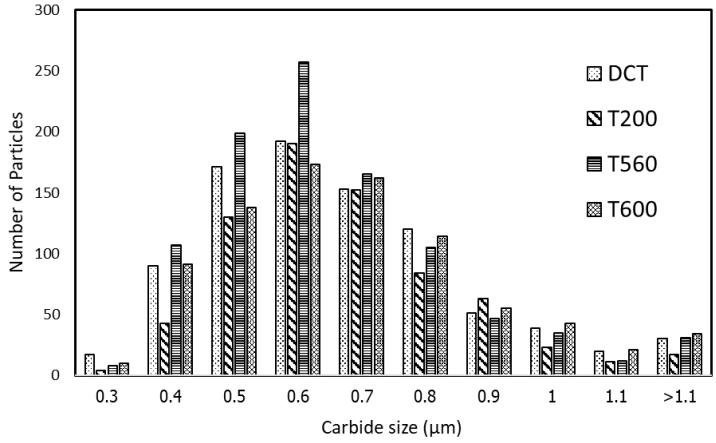

Fig. 7 The MC carbide size distribution measured from SEM micrographs taken at 5000X

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References

[1] T. Arai, Tool materials and surface treatments, Journal of Materials Processing Technology 35 (1992) 515-528. DOI: 10.1016/0924-0136(92)90338-S

[2] Hitachi Metals, Ltd., YSS High Speed Steels. (2015). www.hitachi-metals.co.jp/e/products/auto/ml/pdf/hsts_b.pdf (accessed 31 May 2020)

[3] A. P. Gulyaev, Improved Methods of Heat Treating High Speed Steels to Improve the Cutting Properties, Metallurgy 12 (1937) 65–77.

[4] M. Koneshlou, K.M. Asl, F. Khomamizadeh, Effect of cryogenic treatment on microstructure, mechanical and wear behaviors of AISI H13 hot work tool steel, Cryogenics 51 (2011) 55–61. DOI: 10.1016/j.cryogenics.2010.11.001

[5] A. Akhbarizadeh, M. A. Golozar, A. Shafeie, M. Kholghy, Effects of austenizing time on wear behavior of D6 tool steel after deep cryogenic treatment , Int. J. Iron Steel Res. 16 (2013) 29–32. DOI: 10.1016/S1006-706X(10)60023-4

[6] Y. Dong, X. Lin, H. Xiao, Deep cryogenic treatment of high speed steel and its mechanism, Heat Treat. Met 3 (1998) 55–59.

[7] J. Y. Huang, Y. T. Zhu, X. Z. Liao, I. J Beyerlein, M. A. Bourke, T. E. Mitchell, Microstructure of Cryogenic Treated M2 Tool Steel, Mater. Sci. Eng. A 339 (2003) 241–244. DOI: 10.1016/s0921- 5093(02)00165-x.

[8] D. Das, K.K. Ray, A.K. Duttac, Influence of temperature of sub-zero treatments on the wear behaviour of die steel, Wear 267 (2009) 1361–1370. DOI: 10.1016/j.wear.2008.11.029

[9] K. P. Kollmer, Applications and Developments in Cryogenic Processing of Materials, The Technology Interface 3 (1999).

[10] P. I. Patil, R. G. Tated, A Comparison of Effects of Cryogenic Treatment on Different Types of Steels: A Review, IJCA Proceedings on International Conference in Computational Intelligence (2012) 10–29.

[11] A. M. Russell, K. L. Lee, Structure-Property Relations in Nonferrous Metals, John Wiley and Sons, 2005, p. 18. DOI:10.1002/0471708542

[12] S. Kalia, Cryogenic processing, a study of materials at low temperature, J Low Temp, Phys. 158 (2010) 934–945. DOI: 10.1007/s10909-009-0058-x

[13] A.T. Akono, N. X. Randall, F.-J. Ulm, Experimental Determination of The Fracture Toughness via Microscratch tests: Application to Polymers, Ceramics, and Metals, J. Mater. Res 27 (2012) 485-493. DOI: 10.1557/jmr.2011.402

[14] A. T. Akono, F.-J. Ulm, P. M. Reis, J. T. Germaine, Portable Scratch Testing Apparatus That Can Be Fixed on a Universal Testing Machine, Provisional Patent, Massachusetts Institute of Technology (2013).

[15] A. T. Akono, F. J. Ulm, Fracture Scaling for Scratch Tests of Axisymmetric Shape, J. Mech. Phys 60 (2012) 379-390. DOI: 10.1016/j.jmps.2011.12.009

[16] K. Worasaen, S. Wannapaiboon, K. Tuchinda, P. Suwanpinij, Characterization of secondary carbide in martensitic stainless steel after deep-cryogenic treatment processes, METAL 2019 Conference Proceedings (2019) 575-580. DOI: 10.37904/metal.2019.8310

[17] H. Abrams, Metallography: A Practical Tool for Correlating the Structure and Properties of Materials, ASTM International (1981) p.39. DOI: 10.1520/STP557-EB

[18] B. D. Fahlman, Materials Chemistry, Springer Science & Business Media, 2011, p. 196. DOI: 10.1007/978-94-007-0693-4

[19] F. Meng, K. Tagashira, R. Azuma, H. Sohma, Role of Eta-Carbide Precipitation in the Wear Resistance Improvements of Fe-12Cr- Mo-V-1.4C Tool Steel by Cryogenic Treatment, ISIJ Int. 34 (1992) 205–210. DOI: 10.2355/isijinternational.34.205.

[20] F. C. Campbell, Elements of Metallurgy and Engineering Alloys, ASM International, 2008, p.192. DOI: 10.31399/asm.tb.emea.9781627082518

[21] C. L. Gogte, K. M. Iyer, R. K. Paretkar, D. R. Peshwe, Deep Subzero Processing of Metals and Alloys: Evolution of Microstructure of AISI T42 Tool Steel, Mater. Manuf. Processes 24 (2009) 718–722. DOI: 10.1080/10426910902806210.

[22] P. Paulin, Mechanism and Applicability of Heat Treating at Cryogenic Temperature, Ind. Heat 8 (1992) 24–27.

[23] T. Yugandhar, P.K. Krishnan, C. V. B. Rao, R. Kalidas, Cryogenic Treatment and It's Effect on Tool Steel, 6th International Tooling Conference 24 (2002) 671–84.