Journal of Materials Processing Technology 229 (2016) 398–406

Contents lists available at ScienceDirect

Journal of Materials Processing Technology

journal homepage: www.elsevier.com/locate/jmatprotec

Deep cryogenic treatment of tool steels

B. Podgornik[a,*], I. Paulin[a], B. Zajec[b], S. Jacobson[c], V. Leskovšek[a]

[a] *Institute of Metals and Technology, Ljubljana, Slovenia*
[b] *Hidria Rotomatika d.o.o., Spodnja Idrija, Slovenia*
[c] *Uppsala University, Uppsala, Sweden*

ARTICLE INFO

Article history:
Received 14 April 2015
Received in revised form 26 August 2015
Accepted 27 September 2015

Keywords:
Tool steel
Deep cryogenic treatment
Vacuum heat treatment
Fracture toughness
Load-carrying capacity
Wear resistance

ABSTRACT

The aim of our research work was to investigate the effect of deep cryogenic treatment on fracture toughness, wear resistance and load-carrying capacity of cold work tool steel and to determine the effectiveness of deep cryogenic treatment depending on the tool steel type and chemical composition. The type and chemical composition of the tool steel considerably affect the way how deep cryogenic treatment changes mechanical, tribological and load-carrying capacity of the tool steel. For lower carbon and higher *W* and Co containing cold work tool steel properties can be improved for up to 70%, but are very limited in the case of high-speed steel. At high carbon and vanadium contents properties of cold work tool steels can even be deteriorated after deep cryogenic treatment. In terms of abrasive wear resistance and load-carrying capacity increasing the hardness is the most decisive factor.

1. Introduction

In fine blanking, stamping and punching applications tools are exposed to very demanding contact conditions, including high loads, high contact pressures, elevated contact temperatures and wear. As described by Gåård (2008) tool surface is subjected to complex combination of cyclic mechanical, chemical and tribological loads, which lead to fatigue, chipping and wear of the tool. Ebner et al. (1999) showed that type of tool failure mode and its progression depend on the tool material and heat treatment used, tool shape, design and manufacturing, forming process parameters and work material. However, the biggest impact comes from the tool material and its microstructure. Basic material properties that govern the performance of the tool are hardness, ductility and toughness. Experimental work of Podgornik and Leskovšek (2013) reveals that although the prevention of tool failure is often related to a critical hardness level, the toughness reveals full potential of the material. As the market, especially automotive industry focus toward the use of new light-weight high-strength materials, i.e. high-strength steels (HSS and AHSS), which according to Billur and Altan (2015) are more and more difficult to form, also requirements on tool properties are becoming more demanding. Necati Cora and Koç (2009) point out the importance of different tool properties including hardness, fracture toughness and wear resistance. It also needs to be mentioned, that required tool properties are often not mutually compatible, i.e. high hardness and high toughness.

Traditionally, forming tools are vacuum heat treated in order to obtain microstructure of tempered martensite and uniform distribution of carbides, which gives sufficient fracture toughness at working hardness and acceptable wear resistance. Leskovšek et al. (2006) and Leskovšek and Podgornik (2012) showed that by optimizing heat treatment parameters and using additional heat and thermo-chemical processes tool steel properties and its wear resistance can be enhanced and adjusted for a specific application. In the last years deep cryogenic treatment (DCT) is getting increased attention in many tool applications. It is defined as an add-on process to conventional heat treatment if added after the conventional quenching and tempering process or as supplemental process between quenching and tempering, and involves cooling the material to about $-196\,^{\circ}\mathrm{C}$ for up to 40 h. According to Molinari et al. (2001) cryogenic treatment is not, as often mistaken for a substitute for good heat treatment, but supplemental process to vacuum heat treatment, with the sequence having a considerable influence, as shown by Pellizzari et al. (2008, 2012). Numerous investigations concerning cold-work and high-speed steels have shown that this kind of treatment can lead to improved material performance, including fracture toughness and wear resistance. Review made by Baldissera and Delprete (2008) concludes that the improvement obtained by DCT is mainly contributed to the complete elimination of retained austenite and formation of very small carbides dispersed in the tempered martensitic structure. However, more recent research work of Tyshchenko et al. (2010)

---

* Corresponding author. Fax: +386 1 4701939.
E-mail address: Bojan.podgornik@imt.si (B. Podgornik).

http://dx.doi.org/10.1016/j.jmatprotec.2015.09.045
0924-0136/

and Gavriljuk et al. (2013) clearly shows that the full austenite-to-martensite transformation does not occur in high-carbon steels, with martensitic transformation at low temperature being accompanied by plastic deformation of virgin martensite. An important consequence of plastic deformation which causes partial dissolution of carbide particles is the capture of immobile carbon atoms by gliding dislocations and the formation of carbon clusters that can serve as sites for nucleation of fine η-carbide particles during subsequent tempering. Villa et al. (2014) also showed that in addition to a reduction in the content of retained austenite in high-carbon steels, sub-zero treatment can also improve the stability of the remaining austenite regions against transformation. Interestingly, it was found that while the stability of austenite vs. martensite formation in sub-zero treated tool steel is augmented by the build-up of compressive stresses in this phase, it reduces its thermal stability against decomposition into ferrite and cementite. As explained by Villa et al. (2014) this is due to increased density of defects in austenite, which favor the decomposition of austenite by easy ferrite nucleation. However, despite all the investigations performed so far there are still many contradictory results, with investigations reporting improved as well as deteriorated wear resistance and toughness properties of cold-work and high-speed steels after deep cryogenic treatment. Oppenkowski et al. (2010) investigated the effect of different influencing parameters including austenitizing temperature, cooling rate, holding time, heating rate, and tempering temperature, which all determine the effect of DCT on final tool steel properties. However, the most significant parameters in terms of mechanical properties are austenitizing and tempering temperatures. A low austenitizing temperature in combination with a high tempering temperature improves fracture toughness, bending strength, elongation at fracture, and deformation work, while the opposite combination, high austenitizing and low tempering temperatures, improves hardness and the wear behavior.

Another way of improving tool wear resistance is application of hard wear resistant coatings. Work performed by Leskovšek et al. (2009) on PACVD coated hot forging dies and Sergejev et al. (2011) on PVD coated punches reveal considerable improvement in tool wear resistance by application of hard coatings. However, although the huge potential and benefits of hard PVD, CVD and PACVD coatings in improving friction properties and wear resistance of contact surfaces have been demonstrated by many investigations and successful industrial application, the majority of forming tools are still uncoated and cutting elements in stamping and punching made from tungsten carbide. The major restriction is often complex shape of the forming tool. Furthermore, as found out by Podgornik et al. (2006, 2011) many commercial hard ceramic coatings show high tendency to galling and limited load-carrying capacity, which greatly restrict the use of hard coatings in forming applications. Load-carrying capacity can be simply improved by increasing substrate hardness, which on the other hand also results in reduced fracture toughness. However, under cyclic loading, typical for many forming applications, resistance to crack initiation and propagation is equally or even more important than coating wear resistance. Podgornik et al. (2015) discovered that substrate properties, especially ductility and fracture toughness have significant effect on wear behavior and load-carrying capacity of coated surfaces.

The aim of our research work was to investigate the effect of deep cryogenic treatment on fracture toughness, wear resistance and load-carrying capacity of cold work tool steel and to determine the effectiveness of deep cryogenic treatment depending on the preceding vacuum heat treatment as well as tool steel type and chemical composition.

Table 1
Nominal chemical composition of tool steels used in the investigation (in wt%).

Material	*C*	Si	Mn	Cr	Mo	*V*	*W*	Co
A1	0.85%	0.55%	0.40%	4.35%	2.80%	2.10%	2.55%	4.50%
A2	2.45%	0.55%	0.40%	4.20%	3.80%	9.00%	1.00%	2.00%
B1	1.65%	0.60%	0.30%	4.80%	2.00%	4.80%	10.40%	8.00%

2. Experimental

2.1. Materials and heat treatment

Reference material used in this investigation was a commercial high fatigue strength *P/M* cold work tool steel (designated A1) with lower *C* and high *W* and Co content. In order to evaluate effectiveness of deep cryogenic treatment on fracture toughness and load-carrying capacity two more P/M tool steels were included in the investigation, namely high *C* and *V* content cold work tool steel (A2) and one high-speed steel (B1). Chemical compositions are given in Table 1. After specimens (Plates $20 \times 20 \times 8$ mm, cylinders $\varphi 10 \times 100$ mm and circumferentially notched tensile bar specimens—CNPTB, described by Podgornik et al. (2014a)) were machined from soft annealed blocks, they were vacuum heat treated in a horizontal vacuum furnace with uniform high-pressure gas-quenching using nitrogen gas at a pressure of 5 bar. In order to evaluate the effect of preceding vacuum heat treatment three sets of vacuum heat treatment conditions, resulting in different hardness and fracture toughness combinations were used and combined with deep cryogenic treatment (Table 2). Heat treatment parameters including austenitizing temperature, soaking time and tempering temperature were selected based on the steel producer recommendations, steel composition and results of preliminary investigation performed by Podgornik et al. (2014b). First group of specimens aimed at obtaining maximum hardness was quenched from high austenitizing temperature and triple tempered for 2 h at lower tempering temperature. To obtain high fracture toughness at working hardness of about 64HRc austenitizing temperature for the second group of specimens was reduced and tempering temperature changed according to Table 2. The third group was quenched from the lowest austenitizing temperature and tempered at increased tempering temperature, which should result in maximum fracture toughness. When combined with deep cryogenic treatment quenching was followed by a controlled direct immersion of the test specimens in liquid nitrogen (LN) at a cooling rate of 5 °C/s. Specimens were then kept in LN for 25 h, left to reach room temperature after extraction from LN and finalized by a subsequent 2 h single tempering (Table 2). The same tempering temperature was used as in the case of conventional heat treatment.

2.2. Coating

After heat treatment cylindrical specimens were surface polished ($R_a = 0.05$–0.10 μm), sputter cleaned and coated with commercial monolayer TiAlN coating with a hardness of 3300HV. Coating was deposited at the substrate temperature of ~450 °C with a thickness of ~2 μm using magnetron sputtering process. Details of the coating deposition process are described in the work of Miletić et al. (2014).

2.3. Fracture toughness and hardness

Quenched and tempered cold-work and high-speed steels have a high notch sensitivity, which makes it very difficult to apply standard fracture toughness measuring methods on these materials. On the other hand, in the case of non-standard CNPTB specimens (details are given in work of Podgornik et al. (2014a)) fatigue