Materials Science and Engineering A 531 (2012) 119–129

Contents lists available at SciVerse ScienceDirect

Materials Science and Engineering A

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

Vacuum heat treatment, deep cryogenic treatment and simultaneous pulse plasma nitriding and tempering of P/M S390MC steel

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ARTICLE INFO

Article history:
Received 29 July 2011
Received in revised form 14 October 2011
Accepted 15 October 2011
Available online 20 October 2011

Keywords:
High speed steel
Deep-cryogenic treatment
Pulse plasma nitriding
Hardness
Fracture toughness
Wear resistance

ABSTRACT

Vacuum heat treatment, deep cryogenic treatment and pulse plasma nitriding are efficient techniques to improve the properties of tool and high speed steels. Sometimes the influence of subzero treatment could be directly ascribed to a specific metallurgical transformation. It is the case of the transformation of retained austenite into martensite, causing a general increase in hardness and higher wear resistance. In other cases, however, the increase in wear resistance is not supported by higher hardness and several theories were proposed to explain the observed results. However, poor experimental evidence was reported in the literature for this phenomenon. Specific attention is paid to the influence of subzero treatment just after quenching and solubilization in the vacuum heat treatment or simultaneous pulse plasma nitriding and tempering (PPNT) cycle of the P/M S390MC high speed steel, respectively. Special emphasis was put on resistance to galling and abrasive wear resistance under dry sliding conditions. From obtained results it can be concluded, that the application of deep-cryogenic treatment results in a significantly higher wear resistance of high speed steels, but no significant improvements in fracture toughness have been noticed.

1. Introduction

In metal forming industries tools can be exposed to very complex conditions, which are the result of different effects (mechanical, thermal, chemical or tribological loading) [1] and eventually lead to tool failure. Beside the fact that forming tools can be very expensive, with the tool material representing only a fraction of the tool costs, every interruption of production causes additional loss of income. Therefore, tool materials need to be properly prepared and treated to fulfil many requirements, which, to a certain extent, are not mutually compatible, i.e. high hardness and high fracture toughness. Beside the material's intrinsic properties, tribological properties of the tool surface, including abrasive wear resistance, coefficient of friction and resistance to galling, will also determine tool's operating lifetime. Using different treatment processes and parameters, the microstructure of a tool steel and therefore its mechanical and tribological properties can be modified and optimized for a selected application [2]. Bearing all discussed things in mind, it is easily realized that general tool steel with the optimum properties' profile is very difficult to create. For that reason over the past few decades, extensive interest has been given to the effect of vacuum heat treatment, deep-cryogenic treatment and pulse plasma nitriding on the performance of tool and high-speed steels [3–6]. From this point of view, the increase of performance, the diminishing of abrasive and adhesive wear as well as galling make the nitriding a valuable process for surface treatment [7–9]. On the other hand, the low-temperature treatment is generally classified as either "cold treatment" or shallow cryogenic treatment (SCT) at temperatures down to about −80 °C (dry ice), or "deep-cryogenic treatment" (DCT) at liquid nitrogen temperature of −196 °C [10]. Cryogenic treatment is not, as often mistaken for, a substitute for good heat treatment, but supplemental process to heat treatment before tempering [7,10]. Although combination of different treatment processes increases tool costs it can greatly improve its performance thus having huge impact on the production.

As reported, the DCT has many benefits. It gives dimensional stability to the material, improves abrasive [10,11] and fatigue wear resistance [12], as well as increases strength and hardness [9,13]. The main cause for this improvement of properties are the complete transformation of retained austenite into martensite and the precipitation of fine η-carbides into the tempered martensitic matrix [10,14–16]. Numerous practical successes of cryogenic treatment and research projects have been reported worldwide [14]. Dong et al. [17] studied the effect of DCT with respect to the microstructure of T1 high speed steels. It was proved that DCT can improve wear resistance with precipitation of nano-sized

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doi:10.1016/j.msea.2011.10.044

Table 1
Vacuum heat treatment, deep-cryogenic treatment and pulse plasma nitriding parameters.

Spec.A/B, ∅ 10 mm × 110 mm	Spec.C/D, ∅ 18 mm × 9 mm	Austenitizing		Deep-cryogenic treatment		Tempering [°C/h]	Nitriding [°C/h]
		Temp. [°C]	Time [min]	Temp. [°C]	Immersion time [h]		
A1	C1	1130	6	–	–	540/540/510/2 h	
A2	C2	1130	6	–	–	540/540/2 h	520/2 h
A3	C3	1130	6	−196	25	540/2 h	
A4	C4	1130	6	−196	25		520/2 h
A5	C5	1130	6	−196	40	540/2 h	
A6	C6	1130	6	−196	40		520/2 h
B7	D7	1230	2	–	–	540/540/510/2 h	
B8	D8	1230	2	–	–	540/540/h	520/2 h
B9	D9	1230	2	−196	25	540/2 h	
B10	D10	1230	2	−196	25		520/2 h
B11	D11	1230	2	−196	40	540/2 h	
B12	D12	1230	2	−196	40		520/2 h

η-carbides in primary martensite. This was observed also by Stratton [18]. Molinari et al. [6] found out that the DCT of quenched and tempered high-speed steel tools increase the hardness, reduces the tool consumptions due to increase of wear resistance and shortens the time of equipment setup. Alexandru and Bulancea [19] have pointed out that cryogenic treatments could represent a useful method to transform retained austenite prior to tempering and to overcome the problems related to austenite stabilization. Molinari et al. [6] proposed that carbide precipitation occurs with a higher activation energy thus leading to a higher nucleation rate which in turn leads to finer dimensions and a more homogenous distribution. Furthermore, some results indicate [20] that more carbides are precipitated from matrix during tempering if DCT treated. A new phenomenon referred to as tempered martensite detwinning occurred in M2 steel, which showed a reduction of twins after soaking at −196 °C for 35 h. Mohan Lal et al. [5] analyzed the influence of cryogenic treatment on T1 type-high speed steel and concluded that the cryogenic treatment at −180 °C, soaking for 24 h, imparts 110% improvement in tool life made of T1 type high-speed steel.

The overview of references from the topic investigated in this paper has shown no attainable articles which treat a simultaneous pulse plasma nitriding and tempering of P/M high-speed steel after vacuum heat treatment and DCT, which can for specific application, substantially improve tool life-time and shorten the time for the equipment set-up. Furthermore, the treatment parameters including cooling rate, soaking temperature and time, heating rate, tempering temperature and time [21] need to be optimized considering the material and application. Finally, reported investigations were mainly focused on material wear resistance, while resistance to galling still needs to be investigated. The aim of our work was to investigate the influence of vacuum heat treatment, DCT and simultaneous pulse plasma nitriding and tempering treatment parameters (treatment time and temperatures) on Rockwell-C hardness, surface hardness $HV_{0.1}$, calculated fracture toughness and tribological performance of powder-metallurgy (P/M) high-speed steel with respect to wear resistance and resistance to galling under dry sliding conditions.

2. Materials and methods

2.1. Material and treatments

In this work, commercial P/M high-speed Böehler steel grade S390 Microclean, delivered in the shape of rolled, soft annealed and peeled bars was used. The steel had the following composition (in wt.%): 1.47% C, 0.54% Si, 0.29% Mn, 0.023% P, 0.014% S, 4.83% Cr, 1.89% Mo, 4.77% V, 10.05% W and 8.25% Co. Specimens in the shape of rods (∅ 10 mm × 110 mm, labelled A/B) and discs (∅ 18 mm × 9 mm, labelled C/D) were cut and machined from bars and surface polished (discs to $R_a \approx 0.01\ \mu m$ and rods to $R_a \approx 0.1\ \mu m$). Rods and discs were heat treated together in a horizontal vacuum furnace with uniform high-pressure gas quenching using N_2 at a pressure of 5 bars. After the last preheat (1050 °C) the specimens were heated (25 °C/min) to the austenitizing temperatures of 1130 °C and 1230 °C, soaked for 6 min and 2 min, and gas quenched to 80 °C, respectively (Table 1).

The specimens were then either double tempered and stress relieved for 2 h, or removed from the furnace for the DCT followed by a single tempering or simultaneous pulse plasma nitriding and tempering for 2 h. Selection of individual treatment parameters was based on the literature review, industrial experiences and preliminary experimental work. The DCT of selected specimens (Table 1) was performed with controlled immersion of individual test specimens in liquid nitrogen for 25 and 40 h, respectively. Thermo chemical treatment (pulse plasma nitriding) of test specimens was performed in a Metaplas Ionon HZIW 600/1000 reactor equipped with a convection heating system and internal gas/water heat-exchanger for fast cooling. Pulse plasma nitriding at 520 °C was applied using 3 hPa pressure and a total gas flow rate of 75 l/h. The gas atmosphere was 25%N_2–75%H_2. Heating to the process temperature took approximately 3 h and pulse plasma nitriding at 520 °C was 2 h, Table 1.

2.2. Hardness and fracture toughness tests

The Rockwell-C hardness (HRc) and Vickers hardness $HV_{0.1}$ were measured on discs (∅ 18 mm × 9 mm) and rods (∅ 10 mm × 110 mm) using a Rockwell, B 2000 and Vickers, Tukon 2100 B hardness machine. A semi-empirical model (1) for evaluation of fracture toughness K_{Ic}, of high-speed steels [22], where the fracture toughness is deduced on the basis of microstructural parameters and several other material properties was used to calculate the fracture toughness:

$$K_{Ic} = 1.363 \cdot \left(\frac{\text{HRc}}{\text{HRc} - 53}\right) \cdot \left[\sqrt{E \cdot d_p} \cdot (f_c)^{-(1/6)} \times \cdot (1 - f_{c>a_{crit}}) \cdot (1 + f_{aust})\right] \quad (1)$$

The above semi-empirical correlation one was derived by taking into account the critical strain criterion [23], the experimentally determined effects of microstructural parameters and hardness, and requires a careful use of units. The constant, 1.363, was obtained by assuming that the modulus of elasticity, E, is expressed in MPa, the mean distance between undissolved eutectic carbides, d_p, in m, the Rockwell-C hardness in HRc units, f_c and f_{aust} as volume fractions of undissolved eutectic carbides and retained austenite,