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# Sub-zero treatments of AISI D2 steel: Part II. Wear behavior

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## ABSTRACT

Wear behavior of AISI D2 steel specimens subjected to varied sub-zero treatments, namely: cold treatment, shallow cryogenic treatment and deep cryogenic treatment have been studied with respect to that of the conventionally heat treated ones. The wear behavior has been assessed by dry sliding wear tests under varying normal loads as well as by detailed characterizations of worn surfaces, subsurfaces and wear debris. The obtained results reveal that sub-zero treatments improve the wear resistance of the selected steel; the degree of improvement varies in the ascending order of cold treatment, shallow cryogenic treatment and deep cryogenic treatment, and is function of normal load. The operative mechanism of wear under the investigated conditions is severe delamination and the process of wear is found to proceed with the formation of white layer followed by its delamination, governed by the associated extent of plastic deformation of subsurface. The wear rates are governed by the type of sub-zero treatments and the wear test conditions, and these are in conformity with the characteristics of the worn surfaces, subsurfaces and generated wear debris. The overall wear behavior of the specimens has been explained on the basis of their microstructural characteristics and hardness values, which are detailed in the companion paper (part 1).

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#### 1. Introduction

Research over the last two decades has indicated that wear resistance (WR) of tool/die steels can be improved substantially by deep cryogenic treatment than that achieved either by conventional heat treatment or by cold treatment [1–5]. However, industrial acceptance of deep cryogenic treatment as an alternative route for enhancement of the service life of components of tool/die steels is still quite limited [6] in spite of the fact that it is an inexpensive, one time permanent treatment that affect the entire cross section of the component unlike coatings [7]. This is primarily due to the fact that neither the degree of improvement in WR achievable by deep cryogenic treatment is well established, nor the governing mechanism behind this improvement is well understood. On one hand, the reported degree of improvement in WR by deep cryogenic treatment over conventional heat treatment is quite arbitrary, as it is found to vary from a few percent to a few hundred percent even for the same material [6,8,9], e.g., Table 1 in ref. [9]. On the other hand, the improvement in WR by deep cryogenic treatment has been attributed to either of the following reasons: (i) reduction of retained austenite ( $\gamma_R$ ) content only [3,10], (ii) reduction of  $\gamma_R$  content and the low temperature conditioning of martensite [4,11], (iii) preferential precipitation of  $\eta$ -carbide rather than the variation of  $\gamma_R$  content [12], (iv) conversion of  $\gamma_R$  to martensite with associated refinement of carbides [13–15], (iv) increased hardness and improved homogeneity in hardness distribution or to the increased toughness [17] and (v) suitable combination of fracture toughness and hardness [18]. The above explanations have been often put forward without sufficient quantitative experimental evidences, and more often without relevant correlation with the wear behavior. The objective of the present study is to resolve the above-mentioned controversies, albeit partially, by systematic investigation of microstructure, hardness and wear behavior of AISI D2 steel specimens subjected to conventional heat treatment and different types of sub-zero treatments.

The sub-zero treatments are commonly classified as cold treatment (223–193 K) and cryogenic treatment (193–77 K) not only based on the temperature range but also on the basis of cooling agent used to achieve these temperature range; i.e., dry ice in the former and liquid nitrogen in the latter [1–5,17,19]. Investigations pertaining to cryogenic treatment are mostly limited to deep cryogenic treatment (113–77 K) with respect to either conventional heat treatment [2,9,11,17,20] or conventional heat treatment and cold treatment [3,7,12,21,22]. However, shallow cryogenic treatment (193–113 K) is of technological importance, because it is economical than deep cryogenic treatment, moreover its temperature range is sufficient to convert all  $\gamma_R$  to martensite [23] which is

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#### Table 1

Summary of the different phase contents and the hardness values of differently treated specimens.

Specimens	Phase content (vol.%)		Vickers hardness values (GPa)			
	Retained austenite $(\gamma_R)$	Primary carbides	Secondary carbides	Tempered martensite	Macrohardness (HV <sub>60</sub> )	Microhardness (HV <sub>0.05</sub> )
Conventionally heat treated	$9.80\pm0.7$	$6.99\pm0.3$	$6.57\pm0.3$	76.64	$7.44 \pm 0.04$	$9.03\pm0.06$
Cold treated	$4.60 \pm 0.5$	$7.10 \pm 0.4$	$7.02\pm0.5$	81.28	$7.63 \pm 0.05$	$9.29 \pm 0.07$
Shallow cryogenically treated	Negligible	$7.03\pm0.6$	$7.48\pm0.4$	85.49	$7.72 \pm 0.06$	$9.42\pm0.06$
Deep cryogenically treated	Negligible	$6.99\pm0.5$	$10.06\pm0.3$	82.95	$8.04\pm0.03$	$10.06\pm0.10$

#### Table 2

Summary of characteristics of secondary carbides of differently treated specimens.

Specimens	Amount (vol.%)		Mean spherical diameter $(\mu m)$		Mean population density (×10 <sup>3</sup> , nos./mm <sup>2</sup> )		Mean in spacing	Mean interparticle spacing (µm)	
	SSCs	LSCs	SSCs	LSCs	SSCs	LSCs	SSCs	LSCs	
Conventionally heat treated	$3.52\pm0.2$	$3.05\pm0.7$	$0.49\pm0.01$	$2.24\pm0.05$	$161\pm12$	$6.4\pm0.5$	13.5	71.2	
Cold treated	$3.82\pm0.3$	$3.20\pm0.6$	$0.42\pm0.01$	$1.98\pm0.06$	$290\pm24$	$7.9\pm0.6$	10.6	59.9	
Shallow cryogenically treated	$3.86\pm0.3$	$3.62\pm0.5$	$0.39\pm0.01$	$1.88\pm0.04$	$337\pm20$	$10.7\pm0.7$	9.7	50.1	
Deep cryogenically treated	$5.62\pm0.3$	$4.44\pm0.2$	$0.36\pm0.01$	$1.66\pm0.03$	$471\pm26$	$16.6\pm0.6$	6.1	35.6	

SSCs: Small secondary carbides; LSCs: large secondary carbides.

the primary aim of sub-zero treatment [1-10]. So far, investigations regarding the effect of different types of sub-zero treatments on the microstructure and mechanical properties of tool/die steels are very limited and incoherent. Collins and Dormer [13] have studied the hardness and impact toughness of conventionally heat treated and different types of sub-zero treated specimens of AISI D2 steel, however, their measurements of abrasive wear rates have excluded cold treated specimen and the microstructural characterization has been limited only to conventionally heat treated and deep cryogenically treated specimens. Earlier, Moore and Collins [21] have reported the effect of temperature of sub-zero treatments on the variation of hardness of Vanadis 4 and AISI D2 and H13 steels; however, they have attempted neither microstructural characterization nor wear behavior. Investigations on the variation of hardness and dimensional stability for AISI D2 steel by Surberg et al. [23] have fairly covered all types of sub-zero treatments; however, they have not reported any wear results. Therefore, it is of scientific as well as technological importance to investigate the effect of different types of sub-zero treatments on wear behavior and its correlation with the microstructure and hardness; which have been done in the present study. The obtained results and their pertinent analyses are presented in two companion papers (parts I and II). This paper (part II) presents the wear behavior of differently heat treated specimens and offers an insight on how the microstructure controls the WR on the basis of in-depth study of worn surfaces, subsurfaces of worn specimens and generated wear debris. The microstructure as well as bulk hardness and apparent hardness of the matrix have been detailed in the companion paper (part I) [24]; where, it has been demonstrated that the sub-zero treatment not only reduces the  $\gamma_{\rm R}$  content but considerably modifies the precipitation behavior of secondary carbides resulting into improved hardness values. The favorable modifications of secondary carbides are shown to be significantly higher in deep cryogenically treated specimens than that in cold treated or shallow cryogenically treated specimens. The microstructural details and hardness values of differently treated specimens presented in part I [24] are briefly summarized here in Tables 1 and 2 for the purpose of correlating the wear behavior with respect to the corresponding microstructure and hardness value.

### 2. Experimental details

The selected steel for this investigation is a commercial AISI D2 tool steel containing Fe-1.49 C-0.29 Mn-0.42 Si-11.38 Cr-0.80

Mo–0.68 V–0.028 S–0.029 P (all in wt.%). Specimen blanks of suitable size were subjected to conventional heat treatment and different sub-zero treatments in separate batches. The conventional heat treatment consisted of hardening and tempering, while sub-zero treatment involves an additional low temperature treatment cycle intermediate between hardening and tempering treatments. The different sub-zero treatments studied in this investigation are cold treatment, shallow cryogenic treatment and deep cryogenic treatment at 198 K, 148 K and 77 K, respectively. The details of heat treatment and the different techniques employed to characterize the developed microstructures and hardness have been reported in the companion paper (part I) [24].

Dry sliding wear tests following ASTM standard G99-05 [25] were carried out on a computerized pin-on-disc wear testing machine (DUCOM: TR 20LE, India) equipped with LVDT sensors for continuous acquiring of height loss and frictional force data. Fig. 1 depicts a photograph of the employed wear testing equipment. Cylindrical specimens of 4 mm diameter and 30 mm length were used as static pins; the rotating counter face was made of WC-coated En-35 steel disc (surface hardness  $\approx$ 17.2 GPa) having roughness value of  $R_a < 0.5 \,\mu$ m. The pins were machined from the heat treated specimens by using wire electro-discharge machining (EDM) keeping its major axis parallel to the longitudinal direction of the as received forged bar. The faces of the (pin) specimens were



Fig. 1. Photograph of wear testing equipment used in the present study.

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