Arch. Metall. Mater. 67 (2022), 1, 127-135

DOI: https://doi.org/10.24425/amm.2022.137481

HAIDONG ZHANG^{©1}, XIANGUO YAN^{©1*}, ZHI CHEN^{©1}, MINNA ZHAO^{©1}, LIANG TANG^{©1}, YUAN GAO^{©1}, FAN LI^{©1}, YAO HUANG^{©1}, JUNJI LI^{©2}

EFFECT OF CRYOGENIC TREATMENT ON WEAR RESISTANCE AND MICROSTRUCTURE OF 42CrMo STEEL

In this work, thermo-mechanically treated 42CrMo steel was subjected to cryogenic treatment conducted by means of orthogonal design method, followed by low-temperature tempering to investigate the effect of different parameters of cryogenic treatment on wear resistance of 42CrMo steel and to optimize parameters of cryogenic treatment for improving wear resistance. The results of hardness test and wear test show that cryogenic treatment significantly improves wear resistance with marginal changes in coefficient of friction and hardness. Specifically, cryogenic temperature has the largest impact on wear resistance of 42CrMo steel, holding time has medium impact, and the parameter of treatment cycles has the least impact. The optimum parameters of cryogenic treatment are -196° C for 12 hours with one cycle for improving wear resistance. The results of scanning electron microscopy (SEM) and X-ray diffractometry (XRD) analysis indicate that marginal changes in hardness and coefficient of friction may be owing to little amount of transformation of retained austenite, and the significant influence of cryogenic treatment on improving wear resistance of 42CrMo steel can be mainly attributed to segregation of carbon atoms promoted by cryogenic treatment resulting in more precipitation of carbides in subsequent tempering.

Keywords: cryogenic treatment; orthogonal design method; hardness; coefficient of friction; wear resistance; microstructure

1. Introduction

The material of 42CrMo steel (GB/T 3077), equivalent to EN 42CrMo4 steel or AISI 4140 steel according to their chemical composition [1], is an ultra-high strength structural alloy steel containing medium carbon content and low alloy content with good combination of strength and toughness, high hardenability and high-temperature strength [2]. Due to its good mechanical properties along with low cost, the steel is widely used in the application of conical picks in China mining industry based on field research, as well as drive gears, main spindle, plastic mold and other key parts [3]. In these applications, 42CrMo steel is usually subjected to conventional heat treatment (CHT) in order to reach good comprehensive mechanical properties. Among them, wear resistance can greatly influence performance, reliability and service life of the steel, and therefore needs further improving.

Cryogenic treatment (CT), also known as cryogenic processing, is a supplementary treatment to conventional heat treatment of heating-quenching-tempering cycle and generally carried out between quenching and tempering [4]. It is the process of cooling materials down to cryogenic temperature from

room temperature, holding it for a period of time at the cryogenic temperature and then warming up to room temperature for the purpose of enhancing physical or mechanical properties of ferrous and non-ferrous materials through changing microstructure of the treated materials [5,6], such as increasing hardness [7], improving wear resistance [8], extending service life [9] or relieving residual stresses [10,11]. Cryogenic treatment is commonly performed at low temperature below -80°C, and the minimum cryogenic temperature is depending on refrigerant, usually -196°C by using liquid nitrogen due to its easy availability and low cost, or even -269°C by using liquid helium rarely seen in studies owing to its expensiveness [12-16]. In the past decades, much research has been conducted to investigate impact of cryogenic treatment on wear resistance of various types of steels and demonstrated that cryogenic treatment can significantly improve wear resistance of cold work steels [17-23], hot work steels [24-29], high-speed steels [30-34], stainless steels [35], bearing steels [36-39], valve steels [40], carburized steels [41,42], nitrided steels [43], coated steels [44] and so on. On account of these, cryogenic treatment could be an effective and promising technology to ameliorate conventional treatment of

^{*} Corresponding author: yan_xg2008@126.com



TAIYUAN UNIVERSITY OF SCIENCE AND TECHNOLOGY, SCHOOL OF MECHANICAL ENGINEERING, TAIYUAN 030024, CHINA

² TAIYUAN UNIVERSITY OF SCIENCE AND TECHNOLOGY, JINCHENG SCHOOL DISTRICT, JINCHENG 048011, CHINA



42CrMo steel to improve wear resistance so as to enhance performance and prolong service life of tools or parts made of the steel.

To the author's knowledge, there is little research focusing on cryogenic treatment of 42CrMo steel or other similar chromium molybdenum alloy steels to investigate effect of cryogenic treatment on their mechanical properties and microstructure. Yao et al. [45] examined the effect of different holding time (1 h, 3 h and 6 h) on hardness and wear resistance of 35CrMoV (GB/T 3077) directly soaked in liquid nitrogen and found that hardness and wear resistance enhanced the most for holding time of 6 h which was attributed to precipitated carbides. Cverna et al. [46] reported that being cryogenically treated at different cryogenic temperatures (-50°C, -100°C and -150°C), AISI P20 (equivalent to 3Cr2Mo in GB/T 1299) [47] improved in hardness and wear resistance the most at -150°C, which was ascribed to the formation of martensitic phase and carbide transformation. Senthilkumar et al. [48] showed that improvement of wear resistance of AISI 4140 steel after cryogenic treatment with holding for 24 h at -196°C was more than that for 12 h or 18 h, whereas no microstructure analysis was carried out. Senthilkumar et al. [49] also showed that hardness of cryogenically treated AISI 4140 increased compared to the untreated, for which transformation of retained austenite to martensite was responsible. Zhirafar et al. [50] revealed that cryogenic treatment improved hardness of AISI 4340 steel due to transformation of retained austenite to martensite. Sahin et al. [51] studied effect of cryogenic treatment on mechanical properties (excluding wear resistance) of SAE 4140 steel at two different cryogenic temperatures (-140°C and −196°C) in liquid nitrogen for 24 h and found that hardness increased by 0.7% or 1.0% respectively, which was explained with the transformation of retained austenite to martensite. However, in the literature, cryogenic treatment parameters of cryogenic temperature and holding time were separately considered rather than fully considered, and the parameter of cycles was not involved, to the author's knowledge. Besides, optimization of these three parameters was not investigated either.

Hence, this work takes full account of cryogenic treatment parameters of cryogenic temperature, holding time and cycles to investigate their influence on wear resistance of 42CrMo steel by employing orthogonal design method, and further to search for optimum parameters for improving wear resistance of the steel. Meanwhile, microstructure are observed by SEM and XRD to investigate influencing mechanism of cryogenic treatment on improving wear resistance of the steel.

2. Experimental

2.1. Materials and preparation

In this work, the raw material of 42CrMo steel bar was used in experiment. Its chemical composition was determined by using an optical emission spectrometer (OBLF QSN 750-II), which is in accord with the specification in Chinese GB/T 3077 standard, as shown in TABLE 1.

TABLE 1 Chemical composition of 42CrMo steel

Element	C	Si	Mn	Cr	Mo	S	P
Raw material	0.4	0.21	0.63	0.99		0.0046	
GB/T 3077	0.38- 0.45	0.17- 0.37	0.50- 0.80	0.90- 1.20	0.15- 0.25	≤0.035	≤0.035

Workpieces sawed from raw 42CrMo steel bar were first thermo-mechanically treated, namely heated up and forged in 1230-870°C, and then immediately quenched at 860°C in oil of 70°C for 30 minutes to harden the forged workpieces. Each of thermo-mechanically treated workpieces was cut in half, one half for cryogenic treatment and the other half for no cryogenic treatment as a contrast. Halves of the thermo-mechanically treated workpieces underwent cryogenic treatment with a variety of experiment parameters described in the next section, which was carried out in a program-controlled cryogenic processor (FAWIP SLX-30) that is capable of controlling cooling, holding and heating process of cryogenic treatment. Finally, the cryogenically treated workpieces and corresponding workpieces without cryogenic treatment were tempered at 200°C for 2 hours to relieve thermal stress. For each cryogenic treatment experiment, specimens for wear test and microstructure test were cut from tempered workpieces with cryogenic treatment on the outer end, and comparison specimens were cut from corresponding tempered workpieces without cryogenic treatment on the outer end. The whole treatment process is illustrated in Fig. 1.

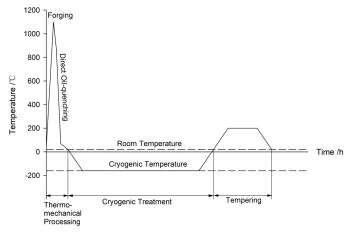


Fig. 1. Treatment processes of 42CrMo steel

2.2. Cryogenic treatment

Cryogenic treatment consists of three stages: cooling down to a cryogenic temperature, holding for a period of time and heating up to ambient temperature, and the following influencing parameters are apparently involved in these stages: cooling rate, cryogenic temperature, holding time, heating rate and cycles (number of treatments), which are varied with different materials in different research studies. In most studies, cooling



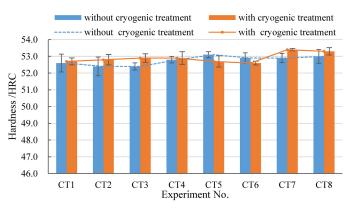


Fig. 2. Hardness of specimens without and with cryogenic treatment

and heating were generally controlled at a slow rate to avoid thermal shock that may result in cracking in microstructure of materials [30,41,51-55], and in addition, the practical cooling and heating rates are mainly around 0.2-2.5°C/min [23,24,35,38-41, 48-54].

In this work, accordingly, cooling rate, as well as heating rate, was set to a slow rate of 2°C/min in order to prevent thermal shock within the workpieces being treated from occurring. The other three parameters of cryogenic treatment – cryogenic temperature, holding time and cycles – which may significantly affect the effect of cryogenic treatment, were selected as major influencing factors of the experiment. In order to investigate influence and optimization of these parameters on wear resistance of the material, the experiment of cryogenic treatment was designed by orthogonal design method by using an orthogonal array to select a representative subset of combinations of the factors at multiple levels of the experiment, which can effectively minimize the number of trials compared to full factorial design of experiment [56]. The factors and their selected levels are listed in TABLE 2, where four levels of cryogenic temperature were

TABLE 2 Factors and levels of cryogenic treatment

Factors	Levels				
ractors	1	2	3	4	
A – Cryogenic temperature /°C	-80	-120	-160	-196	
B – Holding time /h	2	12	_	_	
C – Cycles	1	3	-	_	

 $TABLE \ 3$ $L_8(4^1 \! \! \times \! \! 2^4) \ orthogonal \ array \ of \ cryogenic \ treatment$

Experiment No.	Cryogenic temperature /°C	Holding time /h	Cycles	
CT1	-80	2	1	
CT2	-80	12	3	
CT3	-120	2	1	
CT4	-120	12	3	
CT5	-160	2	3	
CT6	-160	12	1	
CT7	-196	2	3	
CT8	-196	12	1	

selected evenly in the common cryogenic temperature range of -80°C to -196°C , two far different respective levels of holding time and cycles were selected for easily distinguishing their impact. Correspondingly, with the interaction of the factors ignored, the $L_8(4^1\times2^4)$ orthogonal array was suitable for the cryogenic treatment and therefore applied to study the significance of these factors, as shown in TABLE 3. Wear resistance of specimens in wear test was selected as the experiment index to evaluate the effect of cryogenic treatment.

2.3. Hardness and wear test

Rockwell hardness of specimens was determined on the C scale as per Chinese GB/T 230.1 standard by a Rockwell hardness tester (HR-150A) with an accuracy of ± 1.5 HRC, and at least five points on each specimen were uniformly taken to test to average out the hardness value. Wear test was performed at room temperature in dry-sliding condition on a multifunctional tribometer (Zhongke Kaihua CTF-I) with ball-on-disc configuration, and silicon nitride ceramic ball with 5 mm diameter under applied load was pressed against and slid on a rotating specimen for a period of time. The wear test parameters include applied load, contact radius, rotation speed and sliding time, which are shown in TABLE 4, as well as sliding velocity and sliding distance derived from the former parameters. Transient coefficient of friction (COF) at every sampling time (0.1 s) during the wear test was automatically measured and recorded by the apparatus. Wear loss of specimens was weighed on an analytical balance with a range of 10 mg to 200 g and a readability of 0.1 mg.

TABLE 4
Wear test parameters

Applied load	Contact radius /mm	Rotation speed	Sliding velocity /m·min ⁻¹	Sliding time /min	Sliding distance
/1	/111111	/rpm	/m·mm	/111111	/ m
98	13	500	41	60	2450

2.4. Microstructure test

Metallographic specimens for SEM and XRD were both ground with a series of successively finer metallographic sand-papers sticking to a rotating disc and then polished with chrome oxide polishing powder on polishing cloth adhered to a rotating disc. After specimens for SEM were etched in 4% Nital solution for 10 seconds, cleaned with absolute ethanol and dried, SEM test was immediately performed with a scanning electron microscope (JEOL JSM-6510) at magnifications of $5000\times$ and $10000\times$ to examine the morphology of specimens. XRD test was carried out by using an X-ray diffractometer (Rigaku MiniFlex 600) with Cu-K α radiation at voltage of 40 kV and current of 15 mA in order to identify phases in specimens, which were scanned in the angular 2θ range of 35° to 90° .



3. Results

3.1. Hardness

The average hardness with standard deviation of specimens without and with cryogenic treatment of all experiments (CT1 to CT8) is illustrated in Fig. 2, in which lines with markers are also plotted to portray changes and trends in the hardness. The results show that change in hardness of specimens with cryogenic treatment compared to that without cryogenic treatment is very slight – in the range of –0.4 HRC to 0.5 HRC. In view of standard deviation of average hardness, hardness can be considered having no significant change no matter how lower cryogenic temperature is, how long holding time is, and how many cycles are. Overall, these results suggest that cryogenic treatment and its parameters have no significant influence on hardness of the steel.

3.2. Coefficient of friction and wear resistance

The curve of transient coefficient of friction during wear test, taking CT8 as an example, is shown in Fig. 3. In the initial running-in phase, coefficient of friction increases rapidly, and when settling into steady-state phase, it becomes steady. Average coefficient of friction in the latter phase is taken as the coefficient of friction, as shown in Fig. 4. It shows that for every experiment, friction coefficient of specimens with cryogenic treatment has a very slight decrease compared with that of specimens

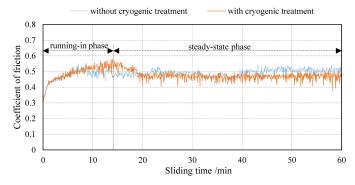


Fig. 3. Transient coefficient of friction of CT8 as a function of sliding time

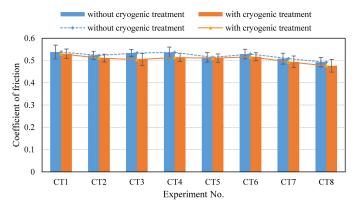


Fig. 4. Average coefficient of friction of wear test

without cryogenic treatment. Taking into account of measuring error, coefficient of friction can be considered having marginal change, in accordance with insignificant change in hardness, which indicates that cryogenic treatment also has no significant influence on coefficient of friction of the steel.

The results of wear resistance of specimens without and with cryogenic treatment are listed in TABLE 5, where relative wear resistance ε and uplift rate of relative wear resistance η are calculated as follows [57]:

$$\varepsilon = \frac{\Delta m_0}{\Delta m_1} \tag{1}$$

$$\eta = \frac{\Delta m_0}{\Delta m_1} - 1 \tag{2}$$

where Δm_0 is wear mass loss of the specimen without cryogenic treatment, Δm_1 is wear mass loss of the specimen with cryogenic treatment.

TABLE 5

Wear resistance of specimens without and with cryogenic treatment (Δm_0 is wear mass loss of the specimen without cryogenic treatment, Δm_0 is wear mass loss of the specimen with cryogenic treatment, ε is relative wear resistance and η is uplift rate of relative wear resistance)

Expenriment No.	$\Delta m_0 / \text{mg}$	$\Delta m_1 / \text{mg}$	$\Delta m_0 - \Delta m_1$ /mg	г	η /%
CT1	35.32	34.99	0.33	1.0094	0.94
CT2	34.45	33.54	0.91	1.0271	2.71
CT3	38.45	37.83	0.62	1.0164	1.64
CT4	35.87	34.52	1.35	1.0391	3.91
CT5	33.80	33.03	0.77	1.0233	2.33
CT6	34.50	31.05	3.45	1.1111	11.11
CT7	32.35	30.77	1.58	1.0513	5.13
CT8	41.59	32.73	8.86	1.2707	27.07

The results presented in TABLE 5 show that wear resistance of specimens with cryogenic treatment is higher than that of specimens without cryogenic treatment by maximum 27.07% in CT8, although hardness and coefficient of friction have no significant change aforementioned, indicating that cryogenic treatment has a significant influence of increasing wear resistance of the steel.

In order to further find the influencing order of cryogenic treatment parameters and to obtain the optimum process of cryogenic treatment, range analysis was performed, and analysis results are shown in TABLE 6, where R_j is the range of characteristic average for a factor j (A, B, or C), K_{jm} is the sum of uplift rate of relative wear resistance (η) at level m for factor j, and \overline{K}_{jm} is mean of K_{jm} .

The result of $R_A > R_B > R_C$ suggests that for wear resistance, cryogenic temperature has the greatest effect, and holding time has medium effect, followed by cycles having the least effect. The highest \bar{K}_{jm} for each factor indicates that the optimum cryogenic treatment is $A_4B_2C_1$, namely $-196^{\circ}C$ of cryogenic



TABLE 6

Range analysis of wear resistance $(R_j \text{ is the range of characteristic average for a factor } j \text{ (A,B, or C)},$ K_{jm} is the sum of uplift rate of relative wear resistance (η) at level m for factor j, and \overline{K}_{jm} is mean of K_{jm})

Factor (j)	A	В	С	
K_{j1}	3.65	10.04	40.76	
K_{j2}	5.55	44.8	14.08	
K_{j3}	13.44	_	_	
K_{j4}	32.2	_	_	
\bar{K}_{j1}	1.825	2.510	10.190	
\bar{K}_{j2}	2.775	11.200	3.520	
\overline{K}_{j3}	6.720	_	_	
\overline{K}_{j4}	16.100	_	_	
Range (R_j)	14.275 8.690		6.670	
Rank order of factors		A > B > C		
Optimum level	A_4	B_2	C ₁	

temperature, 12 hours of soaking time, and 1 cycle of treatment, with the highest wear resistance uplift of 27.07%.

The effects of the major factors on wear resistance are illustrated in Fig. 5. It shows that wear resistance increases with decreasing cryogenic temperature, as well as with increasing holding time, but in contrast decreases with increasing cycles.

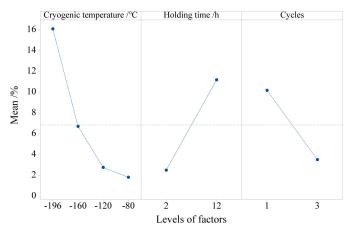


Fig. 5. Main effects of major factors on wear resistance

The variance analysis is performed to determine the contribution of each factor to improving wear resistance, and the results are given in TABLE 7. It shows that cryogenic temperature has the largest contribution of 47.35%, and holding time has the medium contribution of 16.53%, followed by cycles having the least contribution of 8.06%.

Variance analysis of wear resistance

TABLE 7

Factors	A	В	C	Error	Total
Sum of squares	254.87	151.03	88.98	43.36	538.24
Contribution /%	47.35	28.06	16.53	8.06	100.00

3.3. Microstructure analysis

Considering CT8 has maximum improvement of wear resistance, SEM test was carried out on CT8 specimens without and with cryogenic treatment, whose results are shown in Fig. 6. Tempered martensite, a mixture of predominant lath martensite and little plate martensite, can be observed in Fig. 6(a) and (b). A few acicular carbides and granular carbides are also observed in tempered martensite matrix in specimens with and without cryogenic treatment in Fig. 6(c) and (d), and it seems that the amount in the specimen with cryogenic treatment is a bit more than that in the specimen without cryogenic treatment.

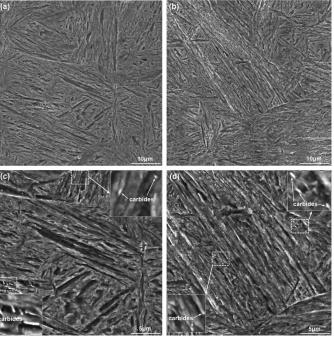


Fig. 6. SEM micrographs of CT8 specimens: (a) and (c) without cryogenic treatment at $5000 \times$ and $10000 \times$ respectively, (b) and (d) with cryogenic treatment at $5000 \times$ and $10000 \times$ respectively

The X-ray diffraction patterns of CT8 specimens without and with cryogenic treatment are shown in Fig. 7. It can be seen that there are characteristic diffraction peaks of martensite (M), clearly indicating the presence of martensite phases, but no characteristic diffraction peak of austenite is detected both in specimens without and with cryogenic treatment, which means that volume fraction of austenite is below the detection limit of the XRD apparatus, approximately 2%-3% [58]. The undetectable austenite can be owed to low carbon content of 0.4% in the steel along with good hardenability of the steel resulting in little retained austenite after quenching.

M(110) peaks of specimens without and with cryogenic treatment are shown in Fig. 8, where peak shifting can be observed. Specifically, the M(110) peak at 2θ of 44.440° of the specimen without cryogenic treatment shifts to 44.559° of the specimen with cryogenic treatment, and correspondingly the d-spacing value of M(110) decreases from 0.20369 nm to 0.20318 nm according to Bragg equation and equation of

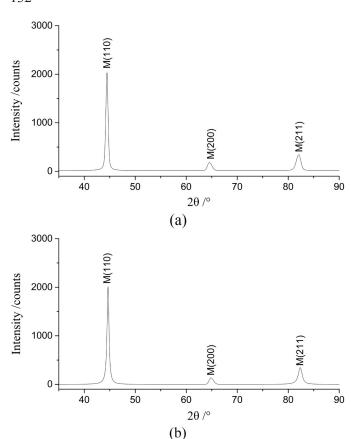


Fig. 7. X-ray diffraction patterns of CT8 specimens: (a) without cryogenic treatment, (b) with cryogenic treatment

interplanar spacing for body-centered tetragonal crystal system [59], indicating contraction of martensite lattice resulting from cryogenic treatment. When calculating d-spacing of martensite lattice, systematic errors were greatly minimized or eliminated with function of real-time angle correction in the control software of the XRD apparatus by using an internal standard sample (Si powder) [60-62].

4. Discussion

Many studies over the past decades have demonstrated that cryogenic treatment has beneficial effects on hardness and wear resistance, and two major reasons are involved in the mechanism of cryogenic treatment on improving hardness and wear resistance of steels: retained austenite transformation to martensite, and forming of fine carbides [23,24,30,40,63-67].

The hardness results indicate that cryogenic treatment has no significant effect on hardness of 42CrMo steel, which is different from Zhirafar et al. [50] results with slight increase in hardness (0.9 HRC) of AISI 4340 steel and Senthilkumar et al. [49] results with a higher increase in hardness (1.7 HRC and 5.3 HRC) of AISI 4140 steel resulting from cryogenic treatment. It has been widely reported that cryogenic treatment can promote the transformation of retained austenite into martensite in steels subjected to cryogenic treatment, which is primarily responsible for improved hardness of steels subjected to cryo-

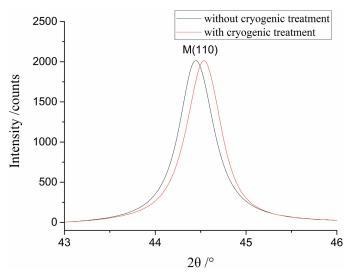


Fig. 8. M(110) peak shifting of CT8 specimens

genic treatment [40,49,50], whereas precipitation of carbides may have little contribution to improved hardness [30]. That is because retained austenite, a soft phase compared with tempered martensite, adversely affects hardness, and therefore a decrease in the amount of retained austenite will lead to an increase in hardness of steels [68,69]. However, no complete transformation of retained austenite could be obtained during cryogenic treatment even treated at liquid nitrogen temperature of -196° C [40,49]. Due to little content of austenite present after quenching in the steel indicated by the XRD results in this study, together with partial transformation of retained austenite caused by cryogenic treatment, little amount of retained austenite transformed to martensite in cryogenic treatment resulting in insignificant change in hardness of the steel.

The results of friction coefficient indicate that cryogenic treatment has no significant influence on friction coefficient of the steel, which is consistent with insignificant change in hardness of the steel affected by cryogenic treatment, and also agrees with Li et al. [33] findings of no obvious change in friction coefficient, but differs from results of Pilla et al. [70] that show a reduction in coefficient of friction led to by an increase in surface hardness of AISI A8 tool steel. It appears that decreasing of coefficient of friction positively correlates with increasing of hardness in cryogenic treatment, and therefore insignificant change in friction coefficient may be correlated to insignificant change in hardness in cryogenic treatment.

Even though cryogenic treatment did not influence hardness and friction coefficient in the steel with little amount of retained austenite present, it still could increase wear resistance of the steel [71]. Results of improved wear resistance by cryogenic treatment in this study are in good agreement with findings of a variety of recent literature [8,17-22,24-42,45,46,48, 57,65,66,72-74] showing that cryogenic treatment can significantly improve wear resistance of steels in different degrees, which is mainly related to cryogenic temperature and holding time. For cryogenic temperature commonly ranging from -80° C to -196° C with a certain holding time, wear resistance



has an increasing trend with lowering cryogenic temperature and reaches the maximum at −196°C if involved in literature [17,24,25,30,33,41,67]. Somewhat differently, increasing holding time varying between 0 h and 132 h at a certain cryogenic temperature (mostly -196°C) increases wear resistance at first [19,31-33,36,45,48,67-69], but then further increasing holding time, if sufficient holding time involved, decreases wear resistance instead [19,31,32,36,68] with exception of Senthilkumar's findings [48], and thus wear resistance obtains the maximum value at the optimum holding time, which varies with different materials in different literature but mostly between 12 h and 36 h, such as 36 h for AISI D2 steel [68,69], 24 h for AISI M2 tool steel [32], 12 h for AISI D3 die steel [19], 24 h for M2 steel [33], 36 h for AISI 52100 bearing steel at -145°C [36], 24 h for AISI 4140 steel [48], 24 h for AISI 316 [31], etc. Although cryogenic temperature and holding time can give rise to improved wear resistance, it is not clear that to what extent their respective influence is and which is more influential. In this study, the range analysis results of wear resistance show that there is an increasing trend of wear resistance with lowering cryogenic temperature with the optimum cryogenic temperature of -196°C, and so is with increasing holding time with optimum holding time of 12 h. The trends agree with findings of recent literature, so cryogenic treatment can be performed at -196°C for at least 12 h for maximally improving wear resistance of the steel in future research. Moreover, the variance analysis results show that cryogenic temperature is the most important parameter for improving wear resistance in cryogenic treatment, and holding time is the second important one. As for cycles of treatment, the third important influencing parameter, the results show that one cycle of cryogenic treatment improves wear resistance more than three cycles, which can support why cryogenic treatments were generally carried out only once in relevant studies. Therefore, there is no need to consider this parameter in future research.

In this study, due to little retained austenite presence and probably little amount of its transformation, the factor of retained austenite transformation to martensite maybe has very slight or insignificant influence on improving wear resistance. Therefore, the major influencing factor for improving wear resistance could be the segregation of carbon atoms during cryogenic treatment and carbides precipitation in subsequent tempering process, which is implied by contraction of martensite lattice indicated by the XRD results and a small amount of precipitated carbides shown in the SEM results. Specifically, as volume contraction (contraction of martensite lattice) at cryogenic temperature leads to more supersaturated martensite [40,41], the substructure of which consists predominantly of high density of dislocations and may also twins [75], interstitial carbon atoms in martensite lattice under compressive stress are prone to segregate to nearby crystal defects such as dislocations and to form carbon clusters [64,65], which will grow up into nuclei for carbides precipitation during subsequent tempering. It should be noted that no precipitation of carbides happens in the process of cryogenic treatment, which occurs in the process of subsequent tempering instead [65,76]. In tempering, carbon atoms continue to segregate at dislocations as well as grain boundaries forming carbon clusters, and carbides precipitate from supersaturated martensite, resulting in improved wear resistance [8,14,50,63,64,77-80].

In summary, cryogenic treatment of 42CrMo steel can be conducted at -196° C for at least 12 h with one cycle to achieve optimum improved wear resistance because of carbides precipitation induced by cryogenic treatment. By applying cryogenic treatment to conventional treatment, wear of tools and parts made of 42CrMo steel can be reduced during working, and service life of them can be extended as well. In addition, to possibly further improve wear resistance, further studies concerning extending holding time more than 12 h will need to be undertaken.

5. Conclusions

In this work, cryogenic treatment with different parameters was conducted on 42CrMo steel by applying orthogonal design method, and range analysis along with variance analysis was carried out in order to determine the influence of cryogenic treatment parameters on wear resistance of the steel and to optimize parameters of cryogenic treatment of the steel. Moreover, SEM and XRD tests were performed to examine the microstructure changes of the steel. Overall, the following conclusions can be drawn from the results:

- (1) Cryogenic treatment has no significant influence on hardness of 42CrMo steel, probably because little transformation of retained austenite occurred in cryogenic treatment due to little retained austenite presence after quenching in the steel along with inability of complete transformation of retained austenite caused by cryogenic treatment.
- (2) Cryogenic treatment has no significant influence on friction coefficient of 42CrMo steel, which is in accordance with insignificant change in hardness, but significantly improves wear resistance of the steel, which can be attributed to segregation of carbon atoms promoted by cryogenic treatment evolving to precipitation of carbides in subsequent tempering.
- (3) Cryogenic temperature is the most influencing factor for improving wear resistance of 42CrMo steel, contributing 47.35% to the improvement of wear resistance, and wear resistance increases with lowering cryogenic temperature.
- (4) Holding time is the second influencing factor for improving wear resistance of 42CrMo steel, which contributes 28.06% to the improvement of wear resistance. Holding for 12 h improves wear resistance more than for 2 h.
- (5) The least significant factor is cycles, which contributes 16.53% to the improvement of wear resistance. One cycle of cryogenic treatment obtains much improvement of wear resistance compared to three cycles, hence there is no need to consider this factor in future studies.
- (6) The optimum levels of cryogenic treatment parameters for improving wear resistance are −196°C for cryogenic temperature, 12 hours for holding time and one cycle,



with maximum improvement of 27.07% in wear resistance. Hence, in future work, cryogenic treatment can be conducted at -196°C for at least 12 h with one cycle to maximally improve wear resistance.

Acknowledgments

This work is supported by National Natural Science Foundation of China (Grant No. 51675363) and Ph.D. Startup Foundation of Taiyuan University of Science and Technology (No. 20182042).

REFERENCES

- [1] G. Totten and R. Colas, Encyclopedia of Iron, Steel, and Their Alloys (Online Version), Boca Raton: CRC Press (2016).
- [2] M. Cheng, Z. Wang, X.P. Qin, Experimental Study about the Effect of Heating Power of Spot Induction Hardening on Material Properties of 42CrMo, in: Advanced High Strength Steel and Press Hardening Proceedings of the 3rd International Conference on Advanced High Strength Steel and Press Hardening (ICHSU2016), World Scientific (2017).
- [3] Z.X. Zhao, Materials for Mechanical Engineering, Xidian University Press, Xi'an (2016) (in Chinese).
- [4] M. Chanda (Ed.), Plastics Technology Handbook, CRC Press (2017).
- [5] G. Hartwig (Ed.), Polymer Properties at Room and Cryogenic Temperatures, Springer Science & Business Media (2013).
- [6] S. Narayanan, K. Gokul Kumar, K. Janardhan Redd, CAD/CAM Robotics and Factories of the Future: 22nd International Conference, 19th-22nd July 2006, Alpha Science Int'l Ltd. (2006).
- [7] W. Chen, W. Wu, C. Li, J. Mater. Eng. Perform. 29, 10-22 (2020).
- [8] K. Amini, A. Akhbarizadeh, S. Javadpour, Mater. Des. 54, 154-160 (2014).
- [9] B. Kursuncu, H. Caliskan, S.Y. Guven, Int. J. Adv. Manuf. Technol. 97 (1-4), 467-479 (2018).
- [10] D.A. College, Y. Zhu, Mater. Sci. Eng. A 722, 167-172 (2018).
- [11] A. Palanisamy, T. Selvaraj, S. Sivasankaran, Arabian J. Sci. Eng. 43 (9), 4977-4990 (2018).
- [12] K. Amini, A. Akhbarizadeh, S. Javadpour, Metall. Mater. Engineering 23 (1), 1-10 (2017).
- [13] G. Prieto, J.E. Ipina, W.R. Tuckart, Mat. Sci. Eng. A-Struct. 605, 236-243 (2014).
- [14] K. Amini, A. Akhbarizadeh, S. Javadpour, Metall. Res. Technol. 113 (6), 611-622 (2016).
- [15] M.C.A. Dumasia, V.A. Kulkarni, M.K. Sonar, Int. Res. J. Eng. Technol. 04(07), 2402-2406 (2017).
- [16] N.S. Kalsi, R. Sehgal, V.S. Sharma, Mater. Manuf. Processes 25 (10), 1077-1100 (2010).
- [17] P. Jimbert, M. Iturrondobeitia, J. Ibarretxe, Met. **8** (12), 1038 (2018).
- [18] S. Kumar, M. Nagaraj, N.K. Khedkar, Mater. Res. Expression 5 (11), 116525 (2018).

- [19] A. Akhbarizadeh, S. Javadpour, K. Amini, Mater. Des. 45, 103-109 (2013).
- [20] K. Amini, A. Akhbarizadeh, S. Javadpour, Mater. Des. **45**, 316-322 (2013).
- [21] K. Amini, A. Akhbarizadeh, S. Javadpour, Int. J. Miner. Metall. Mater. 19 (9), 795-799 (2012).
- [22] D. Das, A.K. Dutta, K.K. Ray, Mater. Sci. Eng. A 527 (9), 2182-2193 (2010).
- [23] D. Das, A.K. Dutta, K.K. Ray, Mater. Sci. Eng. A 527 (9), 2194-2206 (2010).
- [24] T. Shinde. Surf. Eng. 1-9 (2020).
- [25] J. Soleimany, H. Ghayour, K. Amini, Phys. Met. Metall. 120 (9), 888-897 (2019).
- [26] B. Mokarian, K. Amini, H. Ghayour, Transactions of the IMF 97 (3), 121-128 (2019).
- [27] A. Çiçek, F. Kara, T. Kıvak, J. Mater. Eng. Perform. 24 (11), 4431-4439 (2015).
- [28] K. Amini, M. Negahbani, H.G.K. Amini, Metall. Ital. 24 (11), 4431-4439 (2015).
- [29] M. Koneshlou, K.M. Asl, F. Khomamizadeh, Cryogenics **51** (1), 55-61 (2010).
- [30] X.G. Yan, D.Y. Li, Wear **302** (1-2), 854-862 (2013).
- [31] L. Bhaskar, D.S. Raj, Eng. Res. Express 2 (2), 025005 (2020).
- [32] S. Kumar, M. Nagraj, A. Bongale, Arabian J. Sci. Eng. **43** (9), 4917-4929 (2018).
- [33] J.J. Li, X.G. Yan, X.Y. Liang, Wear **376**, 1112-1121 (2017).
- [34] L.P. Singh, J. Singh, Mater. Test. 57 (4), 306-310 (2015).
- [35] G. Prieto, W.R. Tuckart, J. Mater. Eng. Perform. 26 (11), 5262-5271 (2017).
- [36] I. Gunes, A. Cicek, K. Aslantas, Trans. Indian Inst. Met. 67 (6), 909-917 (2014).
- [37] H. Paydar, K. Amini, A. Akhbarizadeh, Kovove Mater. 52, 163-169 (2014).
- [38] R.S. Siva, D.M. Lal, P.K. Nair, Int. J. Miner. Metall. Mater. 21 (1), 46-51 (2014).
- [39] R.S. Siva, M.A. Jaswin, D.M. Lal, Tribol. Trans. 55 (3), 387-393 (2012).
- [40] M.A. Jaswin, D.M. Lal, A. Rajadurai, Tribol. Trans. **54** (3), 341-350 (2011).
- [41] B. Li, C. Li, Y. Wang, Met. 8 (10), 808-820 (2018).
- [42] K. Amini, A. Akhbarizadeh, A. Araghi, J. Balk. Tribol. Assoc. 22 (3), 2335-2345 (2016).
- [43] P. Wach, A. Ciski, T. Babul, Arch. Metall. Mater. 64 (2) (2019).
- [44] M. Sedlacek, B.S. Batic, D. Cesnik, Mater. Tehnol. 53 (4), 565-574 (2019).
- [45] Y. Yao, Y. Zhou, Metals 8 (7), 502 (2018).
- [46] M. Priyadarshini, A. Behera, C.K. Biswas, J. Braz. Soc. Mech. Sci. Eng. 42, 212 (2020).
- [47] Fran Cverna (Ed.), Worldwide Guide to Equivalent Irons and Steels, ASM international, Geauga County (2006).
- [48] D. Senthilkumar, I. Rajendran, Mater. Manuf. Process. 27 (5), 567-572 (2012).
- [49] D. Senthilkumar, I. Rajendran, Int. J. Microstruct. Mater. Prop. 6 (5), 366-377 (2011).



- [50] S. Zhirafar, A. Rezaeian, M. Pugh, J. Mater. Process. Technol. 186 (1-3), 298-303 (2007).
- [51] I. Sahin, G. Hoke, H. Cinici, Mater. Tehnol. 53 (4), 489-494 (2019).
- [52] P. Raja, R. Malayalamurthi, M. Sakthivel, Bull. Pol. Ac.: Tech. 67, 4 (2019).
- [53] M. Arun, N. Arunkumar, R. Vijayaraj, Meas. 125, 687-693 (2018).
- [54] J. Dossett, G.E. Totten, ASM Hand Book 4A: Steel Heat Treating Fundamentals and Process, ASM International, Geauga County (2013).
- [55] S.S. Gill, J. Singh, R. Singh, J. Mater. Eng. Perform. 21 (7), 1320-1326 (2012).
- [56] K. Krishnaiah, P. Shahabudeen, Applied design of experiments and Taguchi methods, PHI Learning Pvt. Ltd. (2012).
- [57] Y.J. Ma, Y. Liu, B.B. Wu, Heat Treat. Met. 44 (01), 40-44 (2019). (in Chinese)
- [58] J.C. Lindon, G.E. Tranter, D. Koppenaal, Encyclopedia of Spectroscopy and Spectrometry, Elsevier Science, London (2010).
- [59] Marc De Graef, Michael E. McHenry, Structure of Materials: An Introduction to Crystallography, Diffraction and Symmetry, Cambridge University Press (2012).
- [60] R.E. Dinnebier, S.J.L. Billinge, Powder Diffraction: Theory and Practice, Royal Society of Chemistr (2015).
- [61] Greg Parker, Encyclopedia of Materials: Science and Technology, Science and technology (2001).
- [62] Rigaku Corporation, Control Software of MiniFlex: MiniFlex Guidance Reference Manual.
- [63] D. Das, A.K. Dutta, V. Toppo, Mater. Manuf. Processes 22 (4), 474-480 (2007).
- [64] S. Li, N. Min, J. Li, Mater. Sci. Eng. A 575, 51-60 (2013).

- [65] S. Li, X. Wu, Mater. Sci. Technol. 31 (15), 1867-1878 (2015).
- [66] V.G. Gavriljuk, W. Theisen, V.V. Sirosh, Acta Mater. 61 (5), 1705-1715 (2013).
- [67] A. Akhbarizadeh, A. Shafyei, M.A. Golozar, Mater. Des. 30 (8), 3259-3264 (2009).
- [68] E. George. Totten (Ed.), Steel heat treatment: Metallurgy and Technologies. CRC press (2006).
- [69] R. Joseph. Davis (Ed.), ASM Specialty Handbook: Tool Materials, ASM international (1995).
- [70] N. Pillai, R. Karthikeyan, Effect of Deep Cryogenic Treatment on AISI A8 Tool steel & Development of Wear Mechanism maps using Fuzzy Clustering, in: IOP Conference Series: Materials Science and Engineering, IOP Publishing (2018).
- [71] D. Senthilkumar, I. Rajendran, M. Pellizzari, J. Mater. Process. Technol. 211 (3), 396-401 (2011).
- [72] J. Sobotova, P. Jurci, I. Dlouhy, Mater. Sci. Eng. A 652, 192-204 (2016).
- [73] D. Das, A.K. Dutta, K.K. Ray, Wear **266** (1-2), 297-309 (2009).
- [74] D. Das, A.K. Dutta, K.K. Ray, Cryogenics 49 (5), 176-184 (2009).
- [75] G. Krauss (Ed.), Steels: Processing, Structure, and Performance, ASM International (2015).
- [76] S. Li, L. Deng, X. Wu, Cryogenics 50 (11-12), 754-758 (2010).
- [77] S. Li, L. Deng, X. Wu, Mater. Sci. Eng. A 527 (29-30), 7950-7954 (2010).
- [78] V.G. Gavriljuk, V.A. Sirosh, Y.N. Petrov, Metall. Mater. Trans. A 45 (5), 2453-2465 (2014).
- [79] Y. Xiao, W. Li, H.S. Zhao, Mater. Charact. 117, 84-90 (2016).
- [80] X. Xie, X.C. Wu, N. Min, Acta Metall. Sinica **51** (3), 325-332 (2015) (in Chinese).