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Influence of Cryogenic Treatment on Microstructure and Properties Improvement of Die Steel

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Abstract

Cryogenic treatment has been increasingly applied to enhance the hardness, antiwear ability and fatigue performance of die steel. On the basis of reading a large number of research papers and references across the world, the author makes a detailed analysis and brief summary of the influence of cryogenic treatment on microstructure after quenching process or quenching plus tempering process, on first and second carbides, on content of retained austenite, on surface hardness, on mechanical properties and antiwear ability of die steels. It's proved that cryogenic treatment on die steel significantly improves its hardness, antiwear capacity and service life. It's the cryogenic process to make die steel have higher hardness, better antiwear ability, better ductility and longer service life because cryogenic process actually has a good influence on die steel of its microstructure, retained austenite volume and amount and size of the second carbide.

Keywords

Die Steel, Cryogenic Treatment, Retained Austenite, The Second Carbide, Antiwear Ability

1. Introduction

It's called as ordinary cryogenic treatment when materials are generally cooled lower than zero degree Celsius, say 0˚C to −130˚C, and stay for a period. But it's called as deep cryogenic treatment if the temperature is even lower, say −130˚C to −196˚C. Deep cryogenic treatment is usually called super low temperature treatment.

2. Cryogenic Treatment's Influence on Microstructure

It's [1] been found that the antiwear ability can be greatly improved if die steel is cooled down to −130˚C or

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even to −196˚C after its quenching. It is because most proportion of retained austenite is transformed to martensite which has a higher hardness and a better antiwear capacity than that of austenite. And some martensite even decomposes to *ε* carbide which is as thin as nano-level article and is also conducive to improvement of antiwear ability after the die steel is tempered.

When X30CrMoN15-1 steel is quenched at 1200˚C for 2 hours in a argon protection furnace [2], retained austenite is observed by optical microscope besides martensite and carbide (see **Figure 1(b)**, marked as A). But when the quenching temperature is lowered to 1000˚C or 900˚C, the retained austenite no longer exists (see **Figure 2**).

Shaohong Li also gets a result concerning the influence of cryogenic treatment on retained austenite volumes and widths of martensite laths [3] (see **Table 1**). **Table 1** expresses that the retained austenite contents is significantly reduced by cryogenic treatment, with 16.9% of retained austenite content at the original quenching state, 4.1% of it after the first cryogenic treatment and 2.5% of it after the second cryogenic treatment.

O. N. Mohanty [4], R. E. Reed-Hill [5], D. V. Edmonds, K. He, F. C. Rizzo [6] and J. Speer, D. K. Matlock [7]

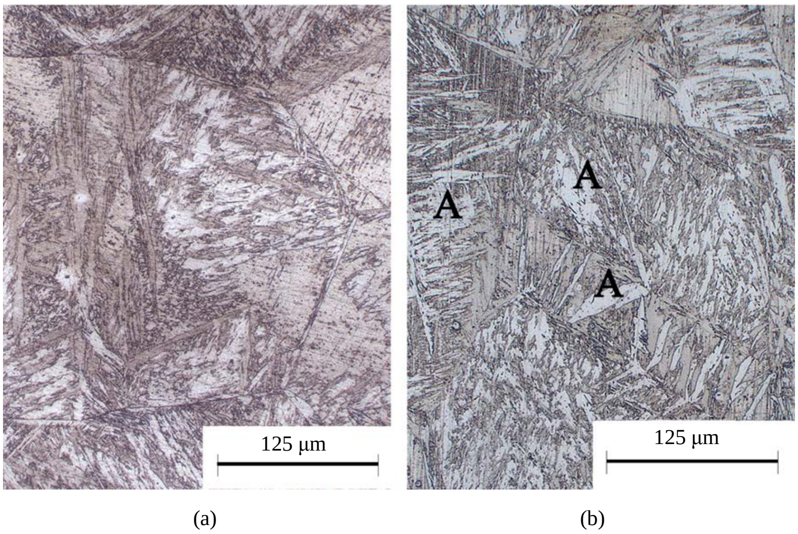

Figure 1. Quenching microstructure of 30CrMoN15-1: martensite + carbide + retained austenite. (a) Quenching in ethylene glycol at 1200˚C; (b) Quenching in air at 1200˚C.

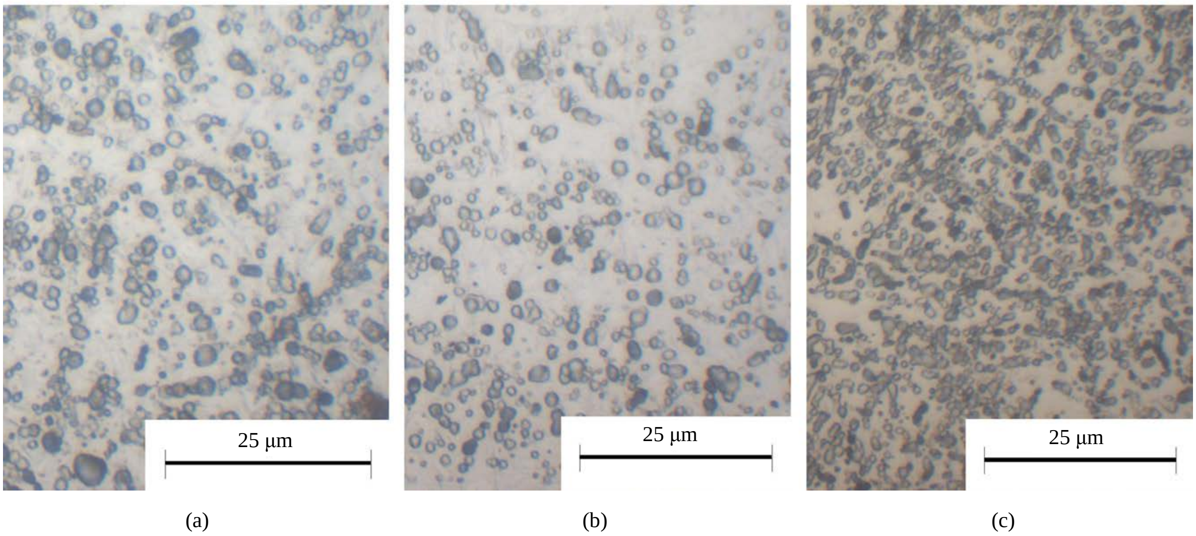

Figure 2. Quenching microstructure of 30CrMoN15-1: martensite + carbide. (a) Water quenching at 1000˚C; (b) Water quenching at 900˚C; (c) Air quenching at 1000˚C.

give full theoretical explanations on how cryogenic treatment reducing the content of retained austenite, facilitating more second carbide to precipitate and increasing the stability of retained austenite so as to improve the antiwear ability and gain a longer service lifetime.

The TEM morphonology [3] of specimens treated as in **Table 1** has been showed in **Figure 3**. Twice cryogenic process makes the martensite laths become thinner than once cryogenic process does, with its width changing from 255 nm for once cryogenic treatment to 60 - 80 nm for twice cryogenic treatment.

3. Cryogenic Treatment's Influence on Mechanical Properties

Das D, Dutta AK, Ray KK [8] [9] contribute the D2 antiwear ability improvement to the scattered distribution of second carbide occurring in case cryogenic treatment.

The austenitizing temperatures have a great influence on retained austenite content and hardness [2] which is displayed in **Figure 4** when the samples are just quenched without a cryogenic treatment. An obvious drop in

Table 1. Influence of cryogenic treatment on retained austenite volumes and widths of martensite laths.

Heat treatment No.	Quenching temperature, ˚C	The first cryogenic treatment, QC	The first tempering, QCT	The second cryogenic treatment, QCTC	The second tempering, QCTCT	Volume percentage of retained austenite	Widths of martensite laths, mm
Quenching	1040˚C/1h					16.9	-
C	1040˚C/1h	−75˚C/2h				4.1	255
D	1040˚C/1h	−75˚C/2h	510˚C/2h			3.3	200
E	1040˚C/1h	−75˚C/2h	510˚C/2h	−75˚C/2h		2.7	76
F	1040˚C/1h	−75˚C/2h	510˚C/2h	−75˚C/2h	510˚C/2h	2.5	66

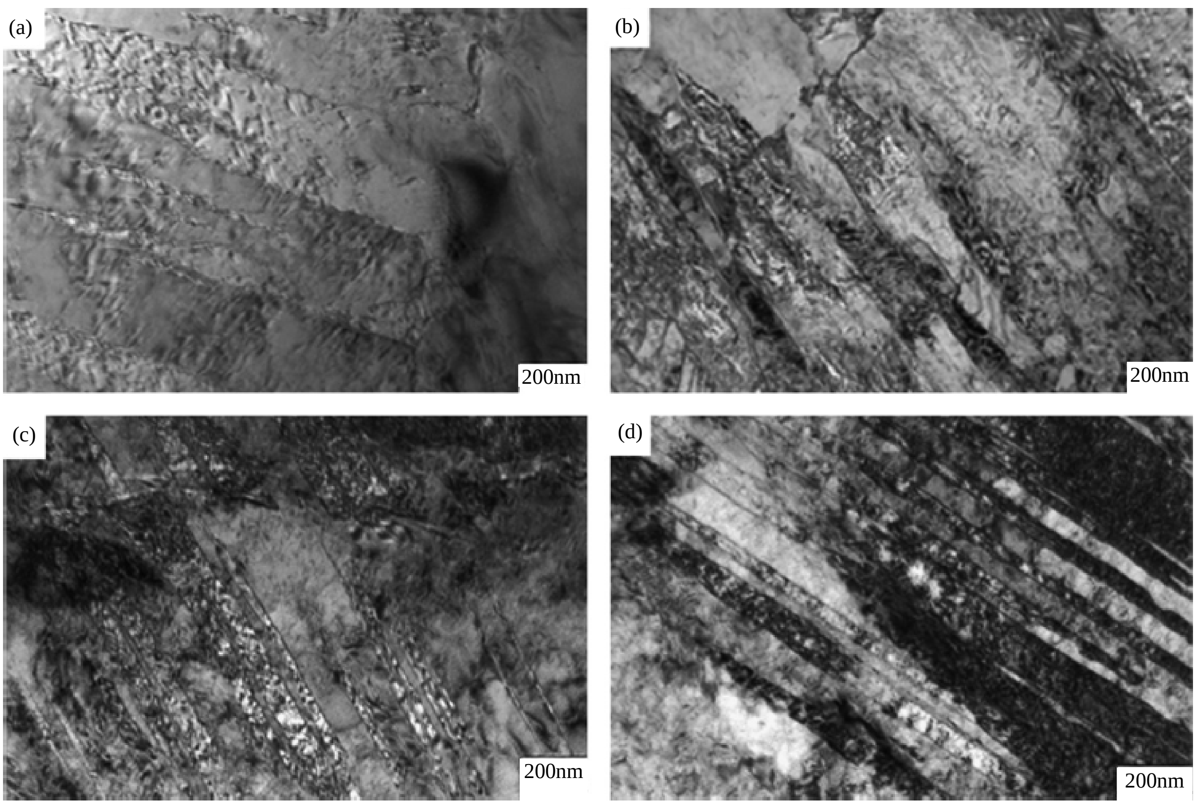

Figure 3. TEM morphology of specimens at different cryogenic treatment. (a) Treatment C; (b) Treatment D; (c) Treatment E; (d) Treatment F.

hardness is seen when the austenization temperatures exceed 1000˚C or even higher, with the highest hardness HRC 56 occurring at 1000˚C and the lowest hardness HRC 33 at 1200˚C. But the retained austenite contents increase steadily while the austenitizing temperatures climb.

However the hardness maintains constant between HRC 54 and HRC 56 even the austenitizing temperature ranges from 900˚C to 1200˚C if the specimens are 24 h permanence at −196˚C just after the quenching process (see **Figure 5**).

The influences [2] of deep cryogenic treatment, which is arranged between quenching and tempering, on hardness and retained austenite content of X30CrMoN15-1 are displayed by **Figure 6** (quenching at 1030˚C) and **Figure 7** (quenching at 1150˚C). The cryogenic temperature is at −196˚C with maintaining period of 24 hours. When quenching at 1030˚C, hardness sees an increase of 10 to 11 HRC after tempering in case of an additional −196˚C cryogenic treatment. The second hardening effect is more obvious in case of cryogenic treatment,

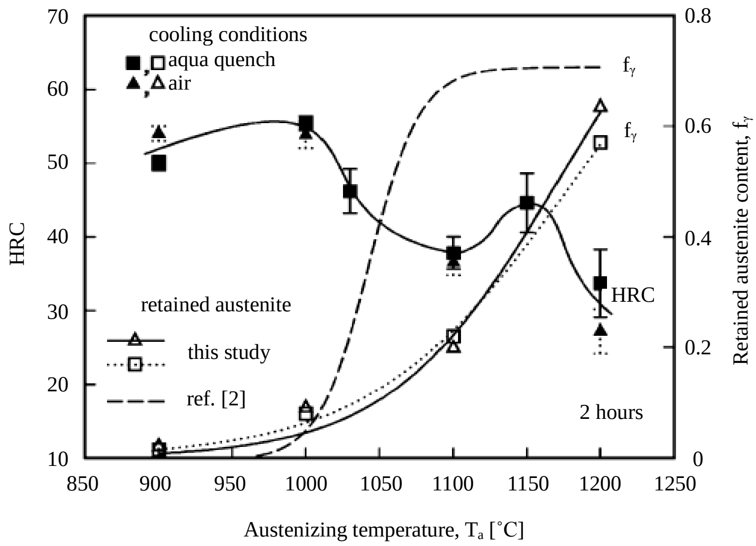

Figure 4. An obvious drop of hardness of X30CrMoN15-1 after the austenitizing temperature exceeds 1000˚C.

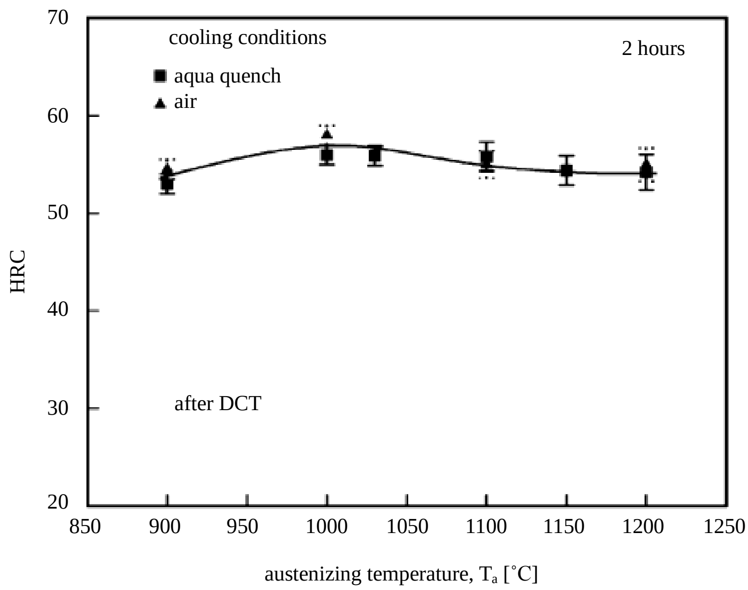

Figure 5. Hardness of X30CrMoN15-1 maintains constant when a deep cryogenic treatment follows after quenching.

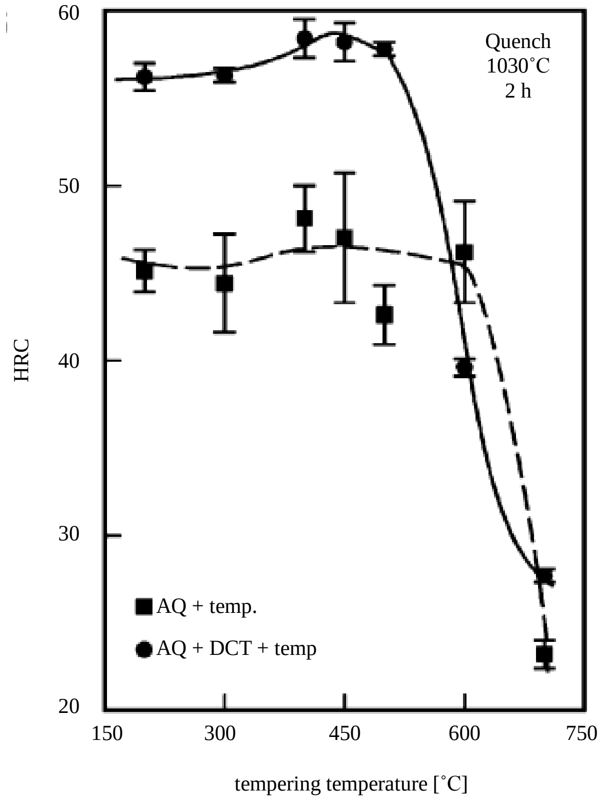

Figure 6. Influence of deep cryogenic treatment on hardness of X30CrMoN15-1 (quenching at 1030˚C).

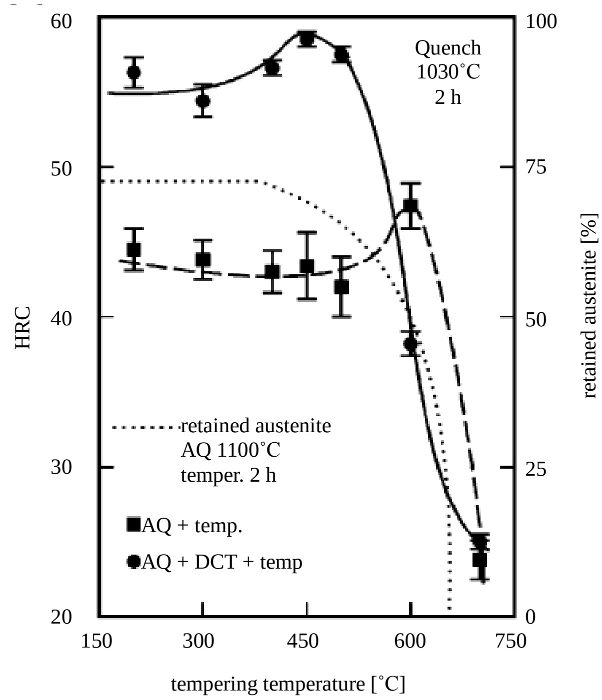

Figure 7. Influence of deep cryogenic treatment on hardness of X30CrMoN15-1 (quenching at 1150˚C).

which facilitates the retained austenite to change to martensite and is conducive to the precipitation of large amount of second fine carbides and carbonitrides from the martensite oversaturated by carbon and nitrogen. Similarly when quenching at 1150˚C, an hardness rise of 11-14 HRC can be obtained.

A simple model is established where the role of martensite, retained austenite, carbide and carbonitride is considered as a whole to set up a function relevance between hardness and quenching temperature. The model is pretty good to predict the volume of martensite, retained austenite, carbide and carbonitride. As a result, the martensite content, carbide as well as carbonitride content reduce with the increasingly higher quenching temperature, but the retained austenite content significantly ascended (see **Figure 8**).

Indeed the mechanical properties of 1.2542 [10] can also be improved through deep cryogenic treatment. In reality, the most exciting point is that the elongation also is raised by 12% - 35% while the tensile strength and hardness are improved by 32% - 36% and 12% respectively when a cryogenic treatment at −196˚C with maintaining periods of 24 - 48 hours after quenching, and tempering at 200˚C is implemented.

4. Influence of Cryogenic Treatment on Second Carbide Sizes and Volumes in Die Steel

It is the large amount of spherical fine second carbide particle precipitated during deep cryogenic treatment that effectively improves the service lifetime and size stability of die steel [11]-[15].

It's been discovered [8] in D2 die steel that the deep cryogenic treatment increases the small second carbide (SSCs) volume amount by 11.4%, increases its distribution density by 250%, decreases its average size by 34%. The deep cryogenic treatment also increases the large second carbide volume amount by 22.3%, increase its distribution density by 100%, decrease its average size by 23% (see **Figure 9** and **Figure 10**).

The finer spheric second carbide particles has been observed [2] clearly through TEM if 1.2542 die steel suffered deep cryogenic treatment. The average diameter of it is 65 nm with soaking temperature −196˚C and tempering at 200˚C (see **Figure 11**).

5. Influence of Cryogenic Treatment on Antiwear Ability

It's been reported [16]-[18] that deep cryogenic treatment definitely greatly improves the antiwear ability of die steel although it does not increase hardness much. Because retained austenite is soft and unstable and easy to change into brittle and hard martensite as well as a change in size, it has a negative influence on antiwear ability in die steel [19].

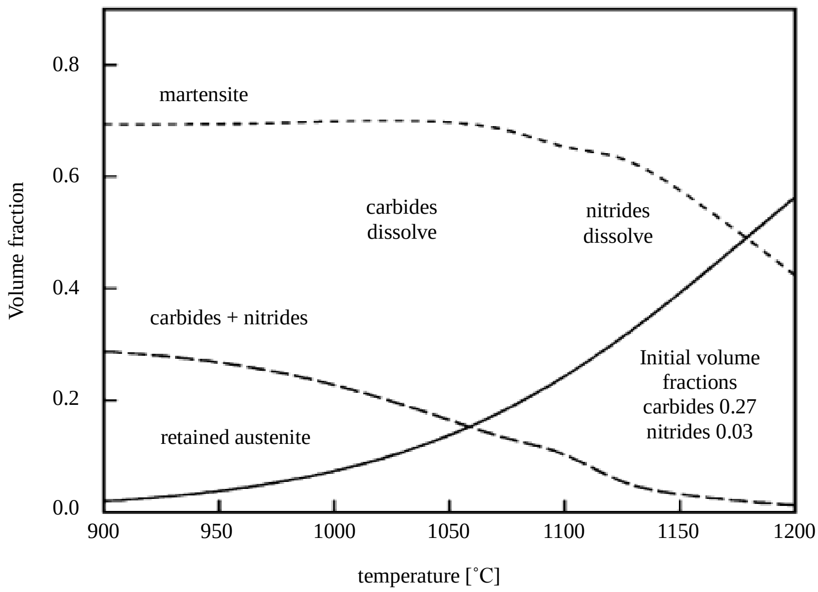

Figure 8. Function relevance between microstructure ingredients of X30CrMoN15-1 and quenching temperatures with deep cryogenic treatment following after quenching.

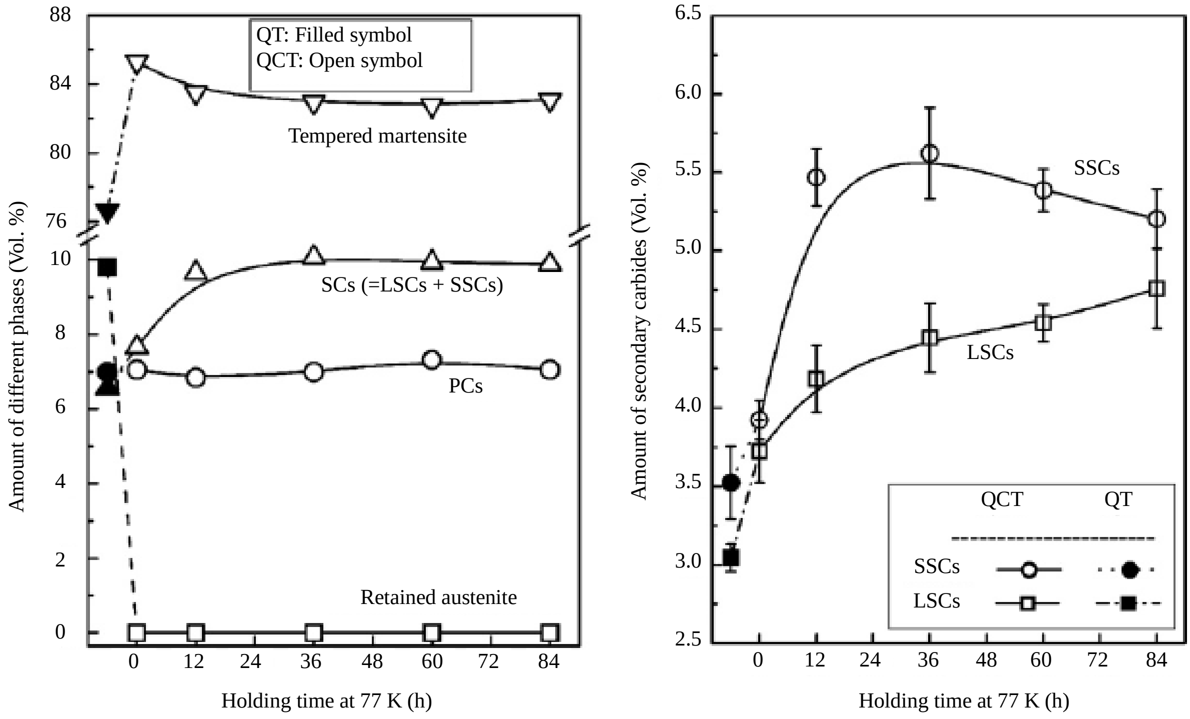

Figure 9. Deep cryogenic treatment influences amount of the small second carbide and large second carbide.

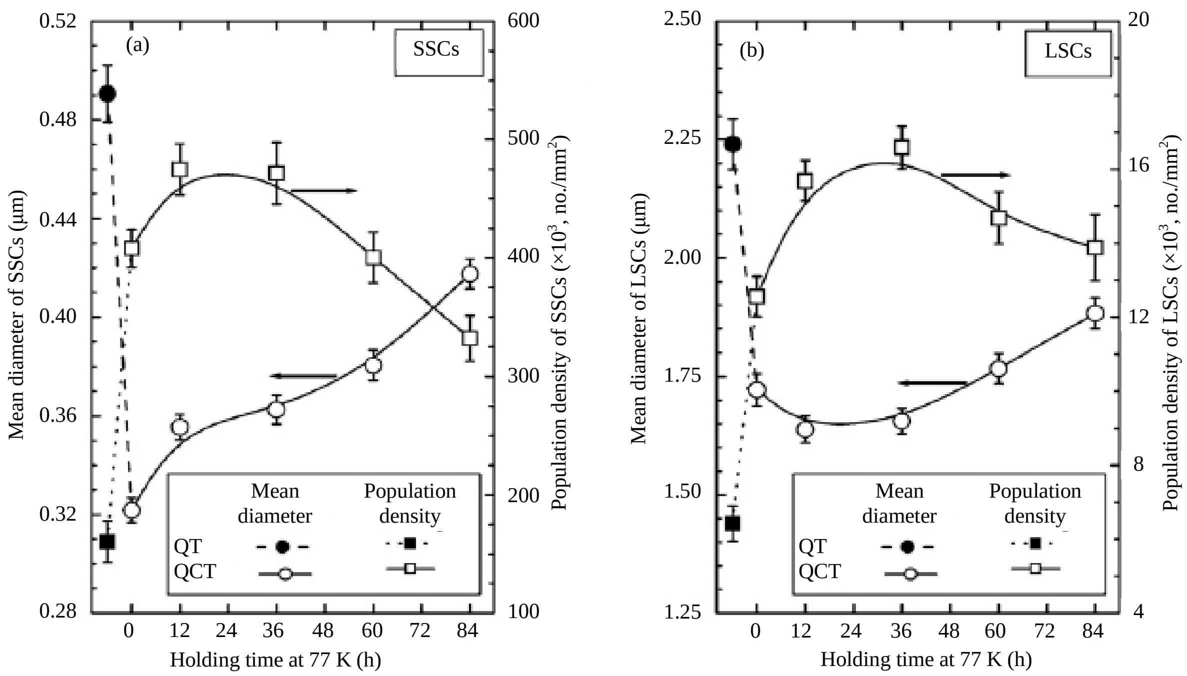

Figure 10. Deep cryogenic treatment influences the sizes and distribution density of the small second carbide and large second carbide.

Influence of cryogenic treatment on antiwear ability in D2 die steel is conducted and the interaction between sliding distance and accumulative wear loss volume is displayed in **Figure 12** [20] [21]. The accumulative wear loss grows rapidly with the die steel in a state of traditional quenching and tempering but it drops dramatically in the case of the die steel experiencing a cryogenic treatment between quenching and tempering.

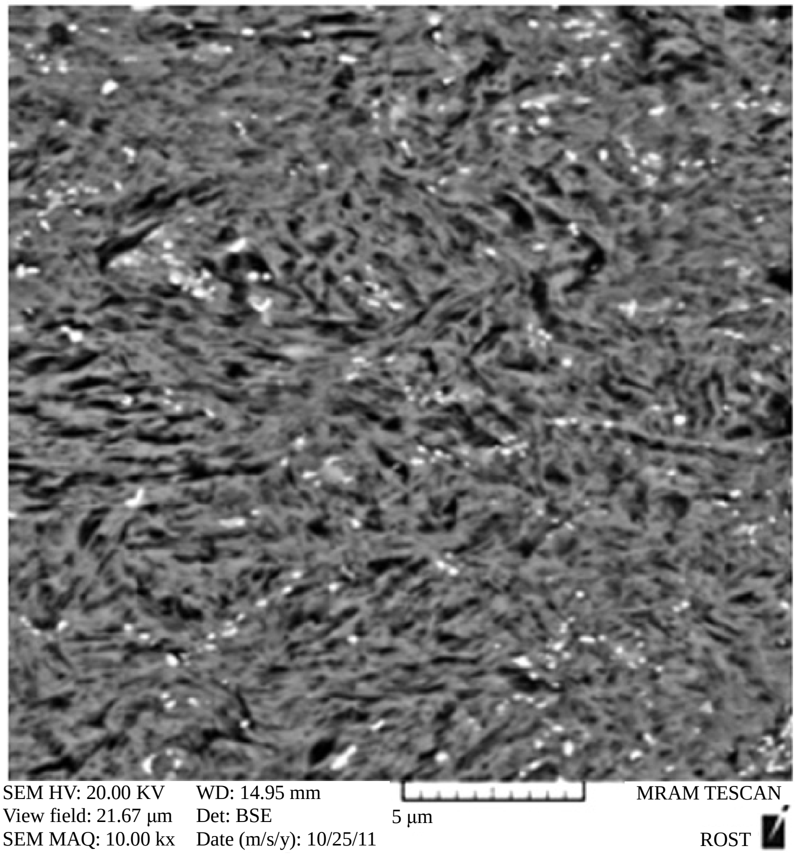

Figure 11. The finer spheric second carbide particles observed in deep cryogenic treatment of 1.2542.

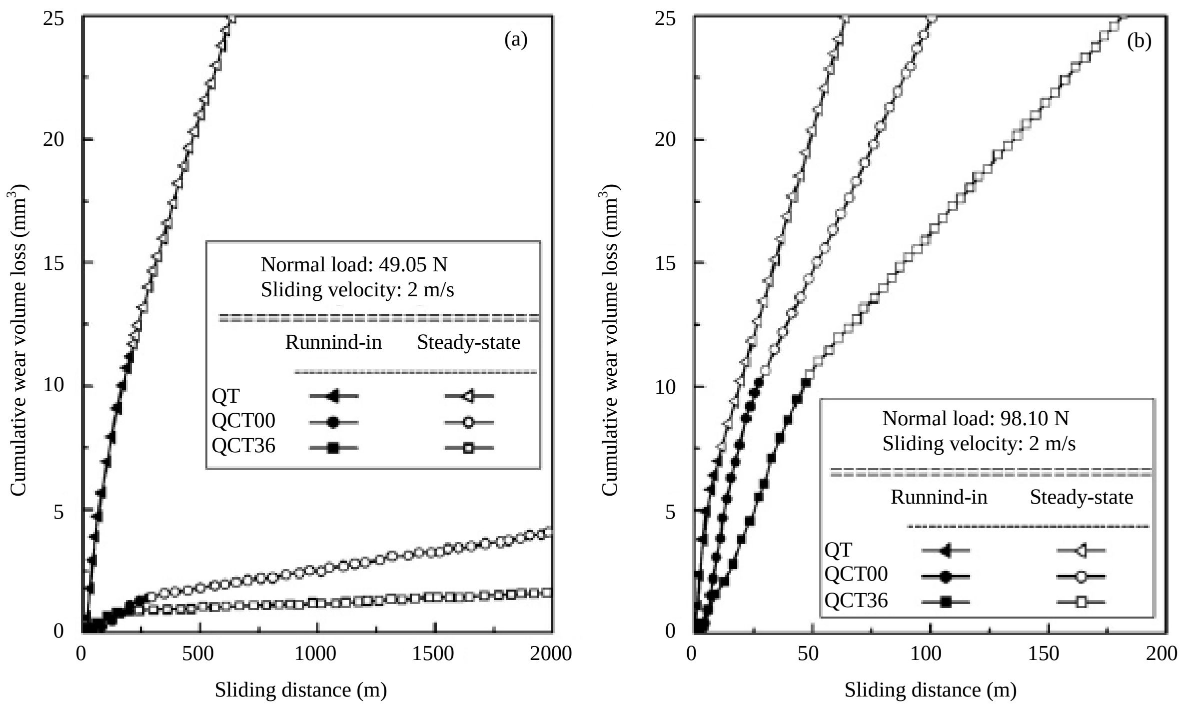

Figure 12. The correlation between sliding distance and accumulative wear volume.

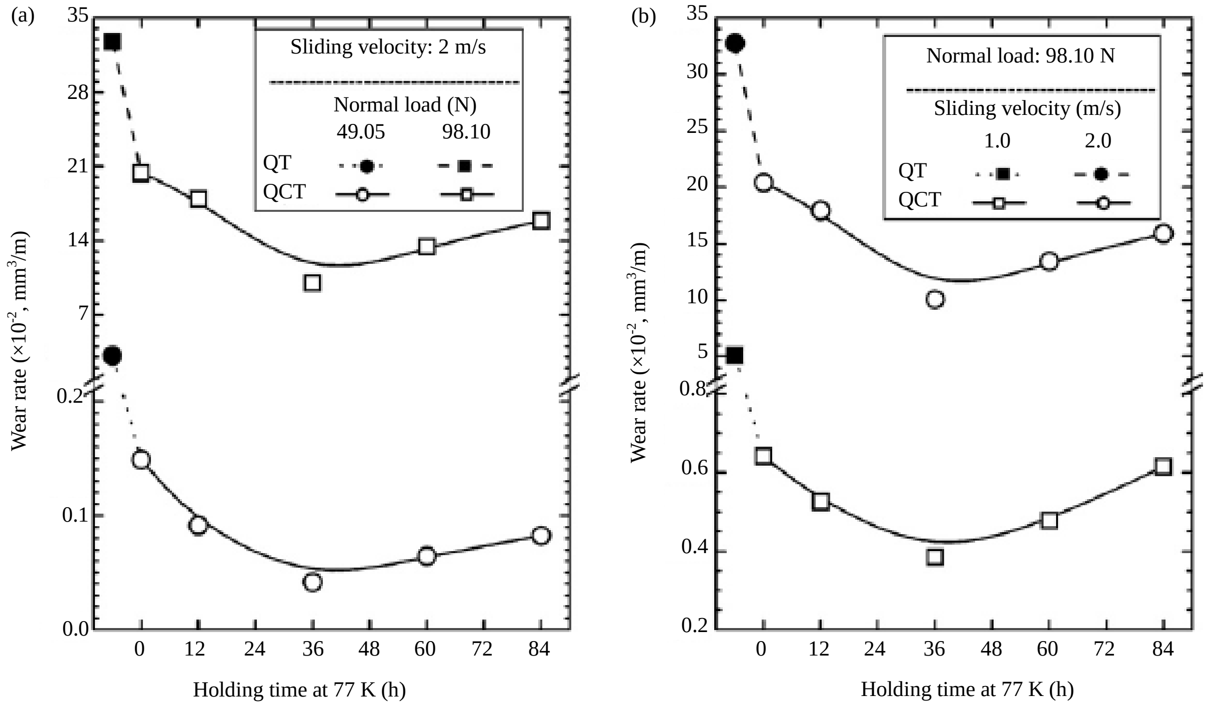

Figure 13. A remarkable benefit on antiwear ability of D2 die steel through cryogenic treatment.

A remarkable benefit on antiwear ability of D2 die steel can be achieved through cryogenic treatment as showed in **Figure 13**. In case of FN = 98.1 N, Vs = 1 m/s and FN = 49.05 N, Vs = 2 m/s, the antiwear ability rises as high as 13.2 times and 76.2 times respectively if there is a cryogenic treatment compared to there isn't.

6. Influence of Cryogenic Treatment on Retained Austenite Content

Study reveals that it is the cryogenic treatment that reduces the volume of retained austenite to improve the size stability and antiwear ability [3]. The large amount of retained austenite 16.9% is tested through X-ray diffraction when the sample just is quenched at 1040˚C with maintenance period of 1 hour. But it is only 4.1% if the sample continues to be cooled down to −75˚C for 2 hours and it continues to drop to 2.5% when the sample experiences twice of cryogenic treatment and twice of tempering.

7. Conclusions

1) Cryogenic treatment increases the mechanical properties, hardness, antiwear ability and service lifetime significantly because it can facilitate the retained austenite to transform to martensite, reduce the volume of retained austenite and improve the stability of retained austenite as well as precipitate more and finer second carbide or carbonitride.

2) Cryogenic treatment helps to reduce the hardness fluctuation range with the variation of quenching temperature as well as increase hardness up to 10 - 14 HRC.

3) It is found that the cryogenic process also reduces the width of martensite laths.

4) Various technological parameters on quenching, cryogenic and tempering process should be adopted for various steel grades to gain best mechanical properties, antiwear abilities and longest service lifetime.

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