

## Cryogenic Treatments of Hot Work Tool Steel (56NiCrMoV7)

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Received on:28 / 6/2010

Accepted on:4/11/2010

### Abstract

The main objective of this paper is to study the effect of Cryogenic treatment of hot work tool steel type (56NiCrMoV7) on the microstructure and mechanical properties. Three different temperature (-50°C,-100°C & -150°C) were selected at different soaking time(1hr,2hr & 3hr) respectively. All cryogenic treatments were adopted after hardening by air & oil respectively. It was concluded that the property enhancement for this tool steel can be attributed by conversion most of retained austenite to martensite accompanied with higher dislocations density that leads to the precipitation of fine carbide during the cryogenic treatment especially at the samples quenched by oil & treated at (-150°C) with soaking 1hr

### أفق جديد في مجال المعاملة الحرارية لصلب العدة نوع 56micrmov7 الساخن

#### الخلاصة

أن الهدف الرئيسي من هذا البحث هو دراسة تأثير المعاملات تحت الصفرية على التركيب المجهرى والخواص الميكانيكية لفولاذ العدة المشكل على الساخن نوع (56NiCrMoV7). أسلوب العمل اعتمد بالأساس على استخدام ثلاث درجات حرارة مختلفة (-50,-100,-150) ° م مع فترات تثبيت زمنية (1,2,3) ساعة متعاقبة لكل درجة حرارة بعد إجراء عملية النقسية بالهواء والزيت.

وخلصت الدراسة الى ان التحسن في الخواص لهذا النوع من الفولاذ يعزى إلى تحول معظم الاوستنايت المتبقي إلى مارتنساييت والمصاحب لحدوث كثافة انخلاعات عالية والتي تؤدي إلى ترسيب لبعض الأنواع من الكاربيدات خلال هذه المعاملة لاسيما للعينات المقاسة بالزيت ولدرجة حرارة (-150) ° م وفترة تثبيت مدة ساعة واحدة.

### Introduction

Heat treatment of tool steel includes hardening and tempering. Cryogenic treatment is modern tempering of tool steel to improve hardness & impact or toughness of parts after quenching. This process typically involves slowly cooling amass of parts to very low temperature & holding then at this temperature for suitable time then slowly heating to ambient temperature.<sup>[1]</sup>

Hot work tool steel already at this stage of design of their chemical compositions anticipated to be subjected to medium & high tempering temperature in order to obtain stable microstructure of stabilized properties during work.<sup>[2]</sup> In (1984), Baron<sup>[3]</sup> studies the effect of cryogenic heat treatment on hardness and the amount of retained austenite, showed the increasing resistance to wear, reduction of internal stresses, consistency of

dimensions and deposition of micro carbides in field is regarded as the most important privileges of using cryogenic heat treatment. The less temperature of cryogenic environment is the more rapid for the improvement in properties is preformed.

In (1994), Meng <sup>[4]</sup> observes precipitation of fine carbides instead of the usual carbides following (-180) °C cryogenic treatment and noted improvement in both resistance and toughness. In (1997), Collins <sup>[5]</sup> discussed the effect of transforming retained austenite to martensite, with the consequently increase in hardness. Deep cryogenic treatment has an effect on martensite; it causes crystallographic and microstructure changes which, on reheating, result in the precipitation of a finer distribution of carbides in the tempering microstructure, with the consequent increase in both toughness and wear resistance.

In (2001), Stajko <sup>[6]</sup> discusses the effect of sub-zero treatment on the wear resistance, toughness and dimensional stability. These properties are critical to the performance of tool steel, wear resistance of resulting tool life of high alloy.

In (2001), Mohanlad <sup>[7]</sup> observes that the lower sub-zero temperature, improve the mechanical properties of hot work tool steel with more obvious as well as tensile strength, toughness and wear resistance. find the original of residual stresses generated due to rolling contact is associated with phase transformation and the cryogenic processing converts the retained austenite into martensite. Therefore cryogenic

processing eliminates the primary mechanism for the generation of residual stresses in both bearing due to rolling contact.

In (2003), Kelkar <sup>[9]</sup> shows that the higher dislocation density in cry treated samples can be attributed to the strain induced by conversion of retained austenite to martensite and different contraction of matrix relative to carbide phase during cooling.

In (2003), Huang <sup>[10]</sup> studies the cryogenic treatment of M<sub>2</sub> tool steel, which involved week-long soaking only carbon clustering effect that resulted in increasing carbide density in the subsequent heat treatment.

In (2003), Bourne <sup>[11]</sup> discusses the wear resistance improvements of D<sub>2</sub> tool steel that varies between a few and a few hundred percent and conflicting results are presented for the change in impact resistance of treated steel.

In (2003), Huang <sup>[12]</sup> identified highly dispersed, nano sized Fe<sub>2</sub>C-carbides in H.S.S cutting tool and in contact to earlier works, found linear proportionality between the cryogenic quenching temperatures and wear rates.

In (2007), Bala <sup>[13]</sup> states that the complexity of phase transformation occurring during tempering of such steels does not always allow to control their properties in such simply way.

In (2008), Bala <sup>[14]</sup> discusses that the decrease of hardness of steel for hot work by increasing the highest temperature of tempering with result of improvement of fracture toughness of each steel.

### Experimental

In this study (56NiCrMoV7) tool steel has been used with different heat treatment sequenced understand the microstructure evolution and the mechanical behavior of the resulted materials. Mechanical testing involved tensile, hardness, impact test. In addition an image analysis was used to evaluate the volume fraction of retained austenite after each treatment schemes. The scheduled process sequence used was illustrated in table (1) for samples quenching in air & table (2) for samples quenching in oil.

This work is accomplished in Al-Sumood Company for steel industries, Nassr Company for mechanical industries, University of Technology Department of Production Engineering & Metallurgy and Ministry of Science & Technology.

### Results & discussion

Fourteenth (14) measurements were taken for each process sequence. Figure (1) shows the plot of hardness as function of the different thermal process sequence in air hardening. The quenching & cryogenic treatment for sample C1, C2, C3 had about (1, 3, 4) HRC higher hardness as compared to B1 while had about (11, 13, 14) HRC as compared to T1. This could be the result from transformation of retained austenite to martensite as showed in table (3). About the samples C4, C5 had slightly decreases in hardness compared with C3, this could be this could be attributed to precipitation of carbide. This effect of hardness can show in

Table (3). Figure (2) shows the plot of hardness as function of the different thermal process sequence in oil hardening. The samples C6,C7,C8 had (1,3,4)HRC higher hardness as compared to the sample B2, While C9,C10 had a(10,12,13) HRC higher hardness as compared to sample T2. This could be the result from transformation of retained austenite to martensite as shown in Table (4). About the samples C9, C10 had slightly decrease in hardness compared with C8, this could be attribution to precipitation of carbide, These effect of hardness can show in Table(3),(4).

Figure (3),(4) show the plot of impact as function of difference thermal sequence , the quench of cryogenic treatment for C1,C2,C3 had (32,39,44)J higher impact as compared to B1 while slightly decrease in impact for sample C4,C5 as compared with C3. This could be attribution to precipitation of dark carbide with along soaking time may explain the reduction in impact & other properties , the similar effect about samples C6,C7,C8,C9& C10 may explain the by the same reason These effect of impact can show in Table(3),(4).

Tensile testing was used to determine the tensile strength of material. Table (5),(6) shows the results of the test for all samples. The highest value of the quenched & cryogenic treatment sample can be attributed to greater initial dislocations

density due to cryogenic treatment, which converts almost all retained austenite to martensite. There is also possibility of strengthening was happened by interaction between dislocations & carbon atoms or alloy elements, which might contribute to the higher tensile strength.

Figure (5),(6),(7) show the relations of hardness, impact & retained austenite for specimens (C1, C2, C3, C4, C5, C6, C8, C9 & C10) respectively.

Figure (7) show the relations of tensile strength, yield strength & elongation for specimens (C1, C2, C3, C4, C5, C6, C8, C9 & C10).

From the results discuss above the following general conclusions can be made. The effects of cryogenic treatment on the microstructure obvious conversion of retained austenite to martensite can be observed in Figure(8), with increase cryogenic temperature increase these conversion to martensite but with long holding time may effect to slightly decrease in property.

The main effect of microstructure results from cryogenic treatment is increase in

the dislocations density resulting from retained austenite conversion to martensite.

**Table (1) represent the groups of samples were treated in air quenching.**

Item	Symbol	Details
1	B1	Specimen's test hardening at (880°C), soaking time (30 min) then quenching in forced air.
2	T1	Specimens test after hardening in forced air, two stage of tempering at (500°C,480°C), soaking (1.5hr) then cooling in still air at each stage.
3	C1	Specimens test after hardening in forced air cryogenic treatment at (-50°C), soaking time (1hr) then heating in still air and then tempering at (220°C),soaking (30min) then cooling in still air .
4	C2	Specimens test after hardening in forced air cryogenic treatment at (-100°C), soaking time (1hr) then heating in still air and then tempering at (220°C),soaking (30min) then cooling in still air.
5	C3	Specimens test after hardening in forced air cryogenic treatment at (-150°C), soaking time (1hr) then heating in still air and the tempering at (220°C),soaking (30min) then cooling in still air .
6	C4	Specimens test after hardening in forced air cryogenic treatment at (-150°C), soaking time (2hr) then heating in still air and then tempering at (220°C),soaking (30min) then cooling in still air.
7	C5	Specimens test after hardening in forced air cryogenic treatment at (-150°C), soaking time (3hr) then heating in still air and then tempering at (220°C),soaking (30min) then cooling in still air.

**Table (2) represent the groups of samples were treated in oil quenching**

Item	Symbol	Details
1	B2	Specimen's test hardening at (860°C), soaking time (30 min) then quenching in oil.
2	T2	Specimens test after hardening in oil, two stage of tempering at (500°C,480°C), soaking (1.5hr) then cooling in still air at each stage.
3	C6	Specimens test after hardening in oil cryogenic treatment at (-50°C), soaking time (1hr) then heating in still air and then tempering at (220°C),soaking (30min) then cooling in still air.
4	C7	Specimens test after hardening in oil cryogenic treatment at (-100°C), soaking (1hr) then heating in still air and then tempering at (220°C),soaking (30min) then cooling in still air.
5	C8	Specimens test after hardening in oil cryogenic treatment at (-150°C), soaking (1hr) then heating in still air and then tempering at (220°C),soaking (30min) then cooling in still air.
6	C9	Specimens test after hardening in oil cryogenic treatment at (-150°C), soaking (2hr) then heating in still air and then tempering at (220°C),soaking (30min) then cooling in still air.
7	C10	Specimens test after hardening in oil cryogenic treatment at (-150°C), soaking time (3hr) then heating in still air and then tempering at (220°C),soaking (30min) then cooling in still air.

**Table (3) results of Hardness, Impact and retained austenite for specimens test Quenching In air.**

No.	Specimens	Hardness			Impact (J)	Retained Austenite (50%)
		H.V	H.R.C	H.B		
1	B1	616	56	578	27	28
2	T1	487	46	435	93	23
3	C1	635	57	598	60	18
4	C2	675	59	636	67	15
5	C3	698	60	657	72	12
6	C4	675	59	636	65	11
7	C5	651	58	610	63	10

**Table(4) results of Hardness, Impact and retained austenite specimens test Quenching in oil.**

No.	Specimens	Hardness			Impact (J)	Retained Austenite (50%)
		H.V	H.R.C	H.B		
1	B2	635	57	598	30	25
2	T2	485	48	452	107	20
3	C6	651	58	610	70	16
4	C7	698	60	657	77	13
5	C8	721	61	673	82	11
6	C9	698	60	657	76	10
7	C10	675	59	636	73	8

**Table (5) results of tensile test for specimens test Quenching In air.**

No.	Specimens	Tensile Strength (Mpa)	Yield Stress (Mpa)	Elongation (%)
1	B1	1274	1093	3
2	T1	1350	1183	15
3	C1	1772	1567	12
4	C2	1943	1763	10
5	C3	2005	1810	8
6	C4	2096	1891	7
7	C5	2188	1954	6

**Table(6) results of tensile test for specimens test Quenching In oil.**

No.	Specimens	Tensile Strength (Mpa)	Yield Stress (Mpa)	Elongation (%)
1	B2	1384	1310	2.7
2	T2	1516	1296	18
3	C6	1980	1798	15
4	C7	2110	1805	13
5	C8	2218	1986	11
6	C9	2399	2028	9
7	C10	2480	2130	8
1	B2	1384	1310	2.7

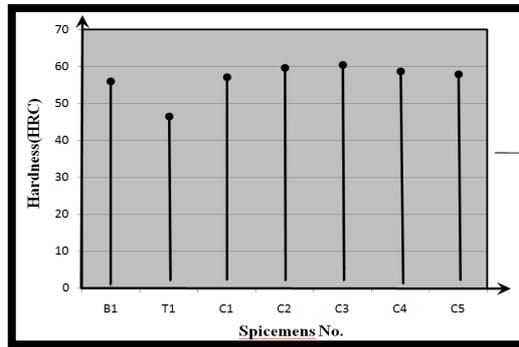


Figure (1) shows the plot of hardness as function of the different thermal process sequence in air hardening.

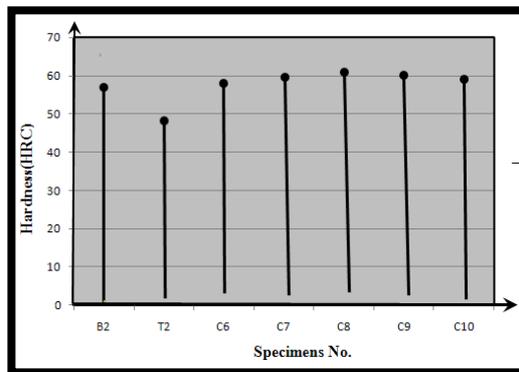


Figure (2) shows the plot of hardness as function of the different thermal process sequence in oil hardening.

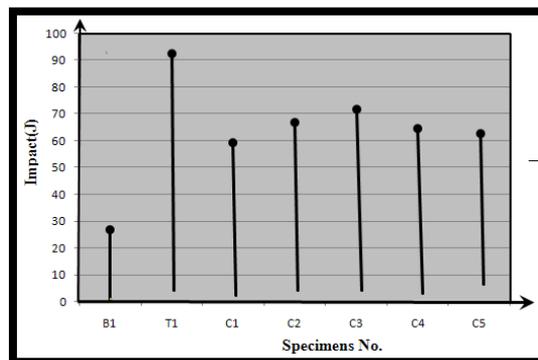


Figure (3) shows the plot of Impact as function of the different thermal process sequence in air hardening.

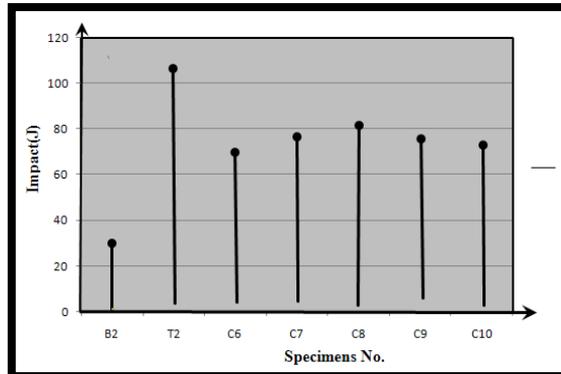
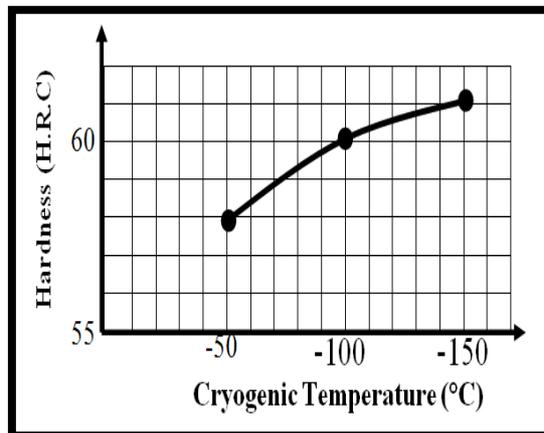
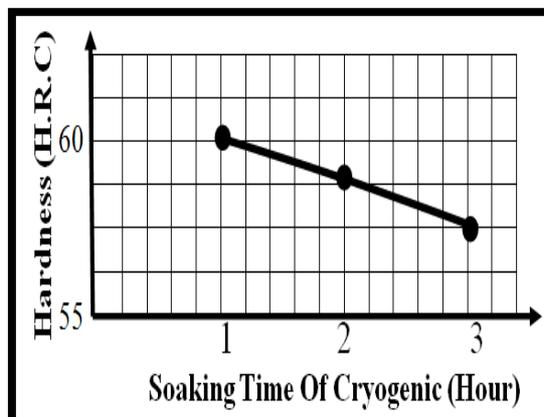


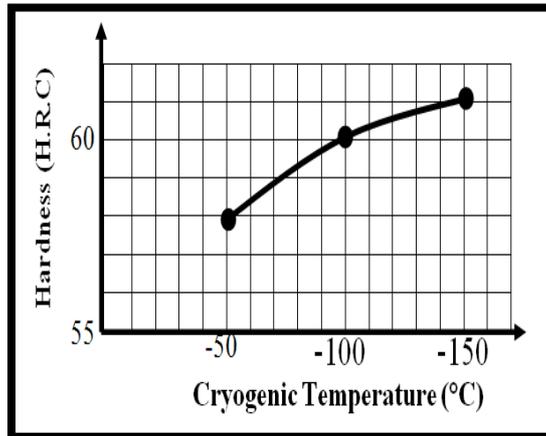
Figure (4) shows the plot of impact as function of the different thermal process sequence in oil hardening



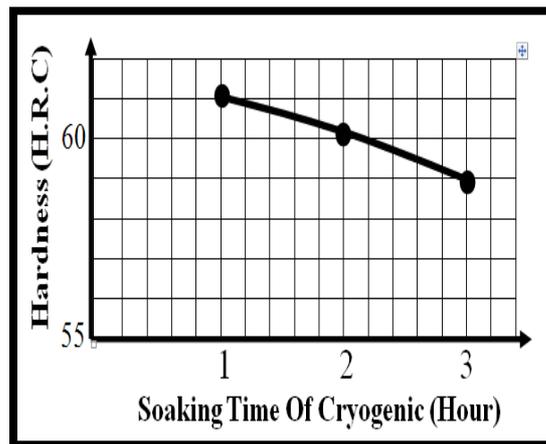
a).show the relation between cryogenic temperature & hardness for specimens (C1, C2 & C3)



b) show the relation between soaking time of cryogenic & hardness for specimens (C3, C4 & C5)

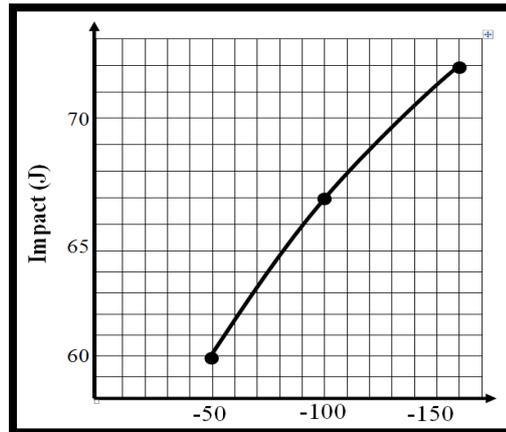


c) show the relation between cryogenic temperature & hardness for specimens (C6, C7 & C8).

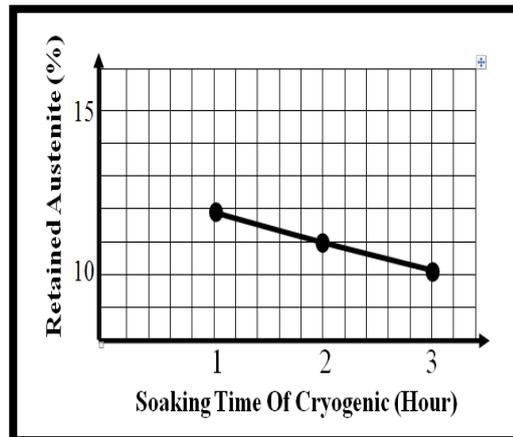


d) show the relation between soaking time of cryogenic & hardness for specimens (C8, C9 & C10).

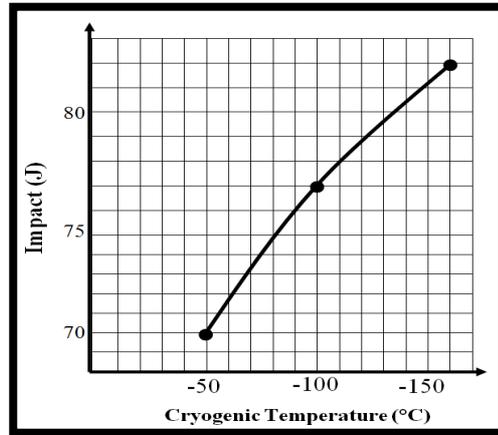
**Figure (5) show the relations of hardness for specimens (C1, C2 , C3, C4, C5, C6,C8, C9 & C10)**



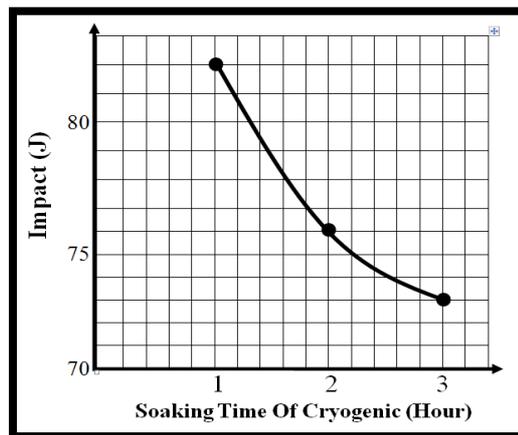
a) show the relation between cryogenic temperature & impact for specimens (C1, C2 & C3)



b) show the relation between soaking time of cryogenic & retained austenite for specimens (C3, C4 & C5)

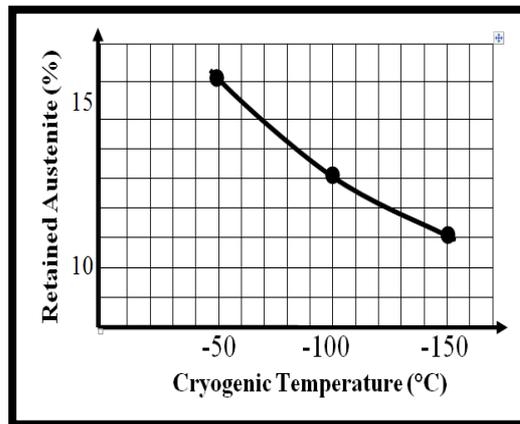


c) show the relation between cryogenic temperature & impact for specimens (C6, C7 & C8).

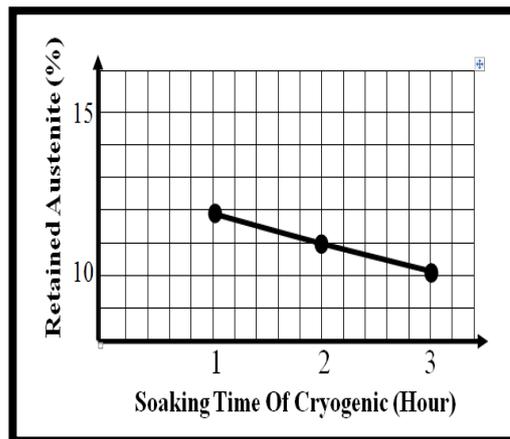


d) show the relation between soaking time of cryogenic & impact for specimens (C8, C9 & C10).

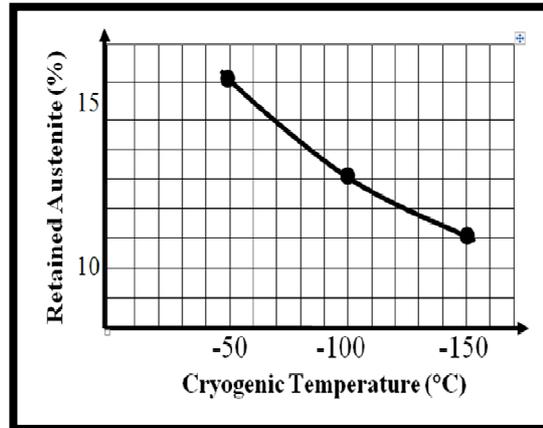
Figure (6) show the relations of impact for specimens (C1, C2, C3, C4, C5, C6, C8, and C9 & C10)



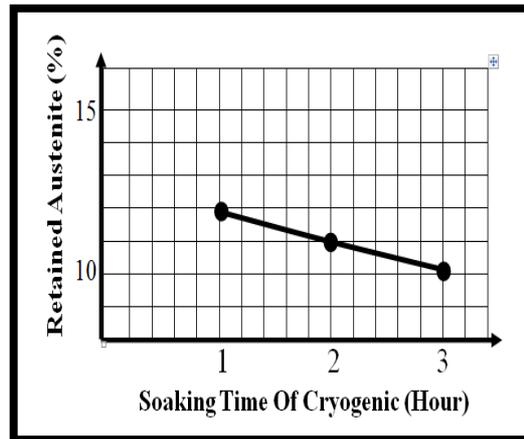
a) show the relation between cryogenic temperature & retained austenite for specimens (C1, C2 & C3)



b) show the relation between soaking time of cryogenic & retained austenite for specimens (C3, C4 & C5)

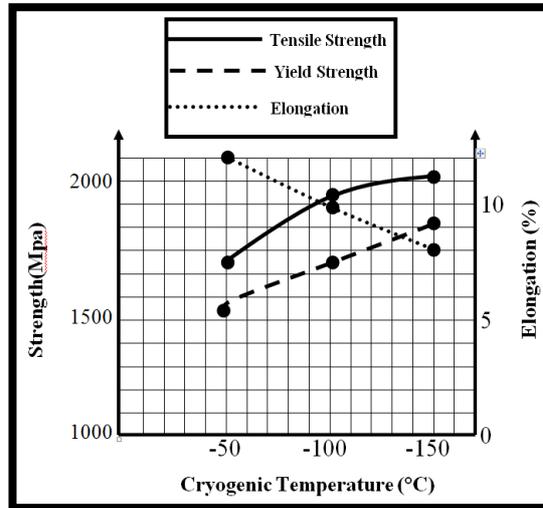


c) show the relation between cryogenic temperature & retained austenite for specimens (C6, C7 & C8).

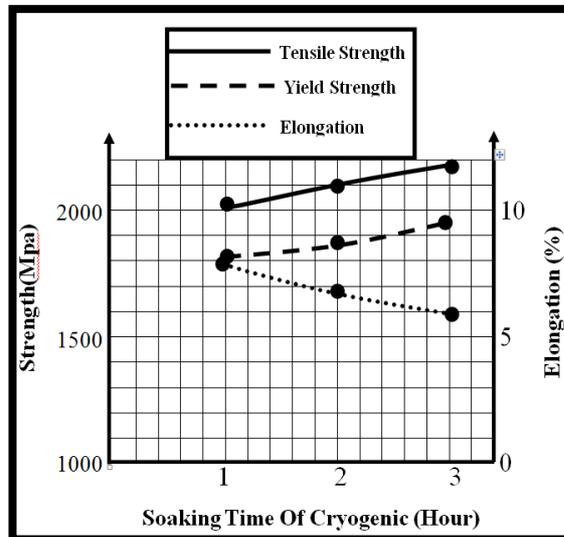


d) show the relation between soaking time of cryogenic & retained austenite for specimens (C8, C9 & C10).

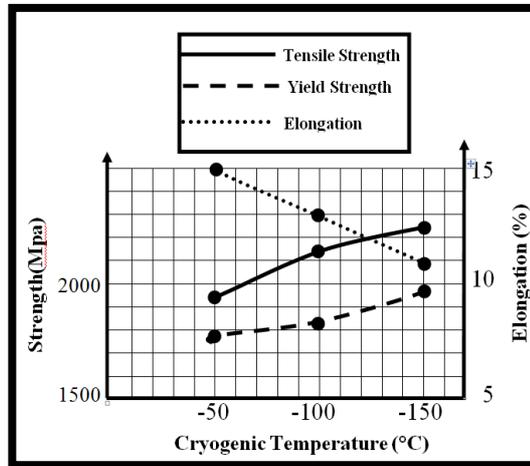
**Figure (7) show the relations of retained austenite for specimens (C1, C2, C3, C4, C5, C6, C8, C9 & C10).**



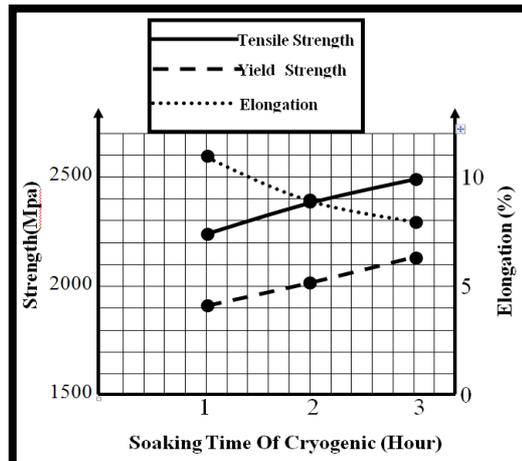
a) show the relation between cryogenic temperature & tensile strength for specimens (C1, C2 & C3)



b) show the relation between soaking time of cryogenic & tensile strength for specimens (C3, C4 & C5)

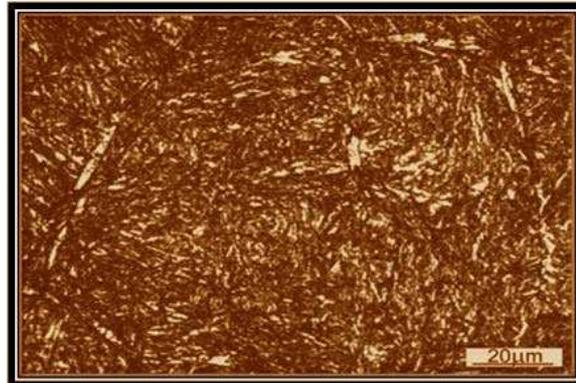


c) show the relation between cryogenic temperature & tensile strength for specimens (C6, C7 & C8).

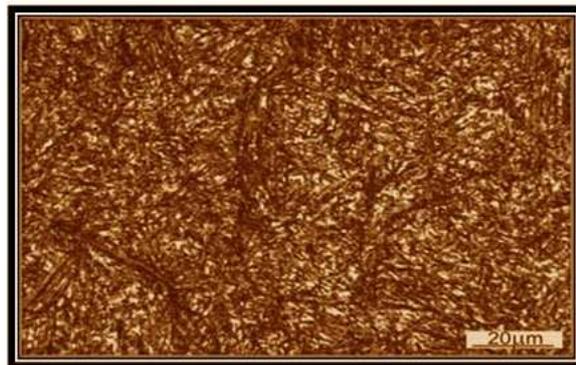


d) show the relation between soaking time of cryogenic & tensile strength for specimens (C8, C9 & C10).

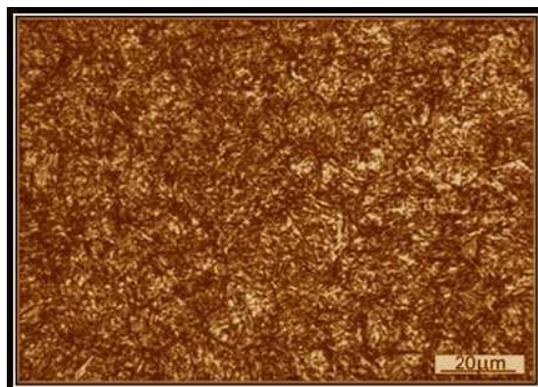
Figure (8) show the relations of tensile strength for specimens (C1, C2, C3, C4, C5, C6, C8, C9 & C10)



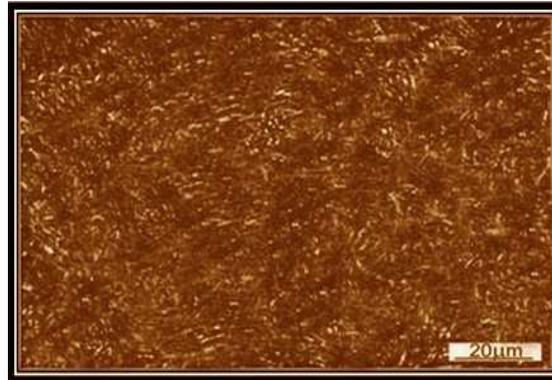
a) show the microstructure for specimen (C1) with retained austenite (18%)  
-1000X



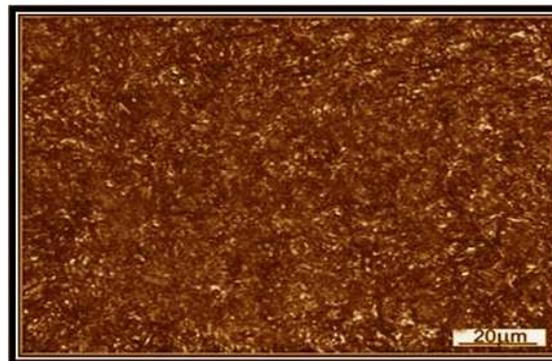
b) show the microstructure for specimen (C2) with retained austenite (15%) -1000X



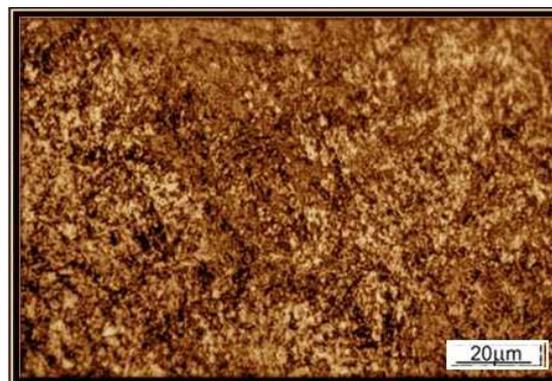
c) show the microstructure for specimens (C3) with retained austenite (12%)  
-1000X



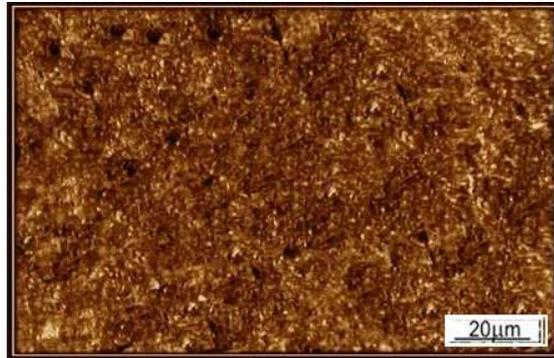
d) show the microstructure for specimen (C4) with retained austenite (11%)  
-1000X



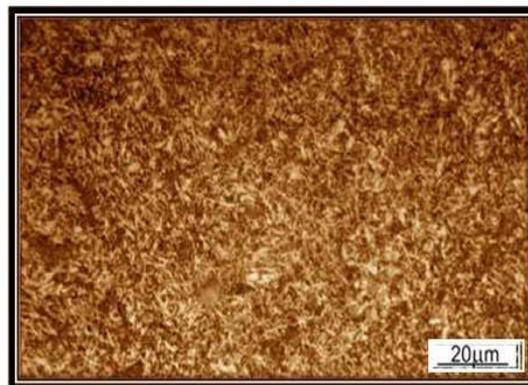
f. show the microstructure for specimen (C5) with retained austenite  
(10%) -1000X



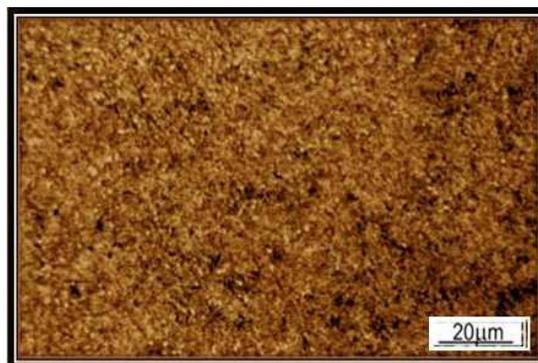
h) show the microstructure for specimens (C7) with retained  
austenite (13%) -1000X



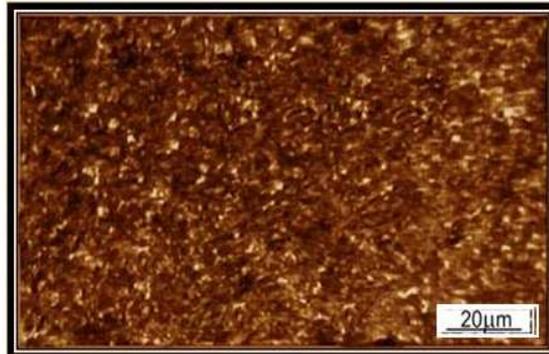
l) show the microstructure for specimen (C9) with retained austenite (10%) -1000X



g) show the microstructure for specimen (C6) with retained austenite (16%) -1000X



k) show the microstructure for specimen (C8) with retained austenite (11%) -1000X



m) show the microstructure for specimen (C10) with retained austenite (8%) -1000X

**Figure (9) show the microstructure of specimens cryogenic treatment.**