# Effect of Cryogenic Trteatment on the Tensile Properties of Carbon Dual Phase Steel

**Ph.D.Abbas Abdul Hussein abid**
University of Baghdad, Mech. Eng. Dept
abbasaltaee@yahoo.com

**Hussein Burhan Muhammad**
Ms.C, Diala University, Mech. Eng. Dept

## ABSTRACT

The aim of this study was to evaluate tensile properties of low and medium carbon ferrite -martensite dual phase steel, and the effect cryogenic treatment at liquid nitrogen temperature (-196 ºC) on its properties. Low carbon steel (C12D) and medium carbon steels (C32D & C42D) were used in this work. For each steel grade, five groups of specimens were prepared according to the type of heat treatment. The first group was normalized, the second group was normalized and subsequently subjected to cryogenic treatment then tempered at (200 ºC) for one hour, the third group was quenched from intercritical annealing temperature of (760 ºC) to obtain dual phase (DP) steel, the fourth and fifth groups were both quenched from (760 ºC), but the forth group was subjected to cryogenic treatment and the fifth group was subjected to cryogenic then tempered at (200 ºC) for one hour. Mechanical tests were carried out which includes, tensile, hardness, as well as microscopic examination. Yield strength, ultimate tensile strength and ductility for DP were correlated to martensite volume fraction. The yield and tensile strength ($\sigma y$, $\sigma u$) of (DP) for the three steels, were higher than those of normalized condition, and increased after cryogenic treatment. These values, for the three steel grades, decreased after tempering at temperature 200 ºC. Tempering of (DP) steel at 200ºC for one hour, after cryogenic treatment, causes the reappearance of yielding point for steels (C12D) and (C32D) while no such a change noticed in (C42D) steel. The results have shown that hardness of (DP) increased after cryogenic treatment for the three steel grades.

**KEY WORDS: Cryogenic treatment، dual phase steel ،tensile properties، hardness ،steel**

# تأثير المعاملة الزمهريرية على خواص الشد للصلب الكربوني ثنائي الطور

## الخلاصة

الهدف من هذه الدراسة هو تقييم خواص الشد للصلب الثنائي الطور المنخفض و المتوسط الكربون ، وتأثير المعاملة الزمهريرية عند درجة حرارة النتروجين السائل (-196 ºC) فى هذه الخواص. استعمل الصلب منخفض الكربون (C12D) و متوسط الكربون (C32D) و (C42D) في هذه الدراسة.قسمت عينات الاختبار لكل صنف من أصناف الصلب على خمس مجاميع وفق المعاملة الحرارية. المجموعة الأولى أخضعت إلى معاملة التطبيع، المجموعة الثانية أخضعت إلى التطبيع ثم المعاملة الزمهريرية ثم المراجعة عند درجة حرارة (200 ºC) مدة ساعة واحدة، المجموعة الثالثة قسيت من درجة حرارة (760 ºC) للحصول على الصلب ثنائي الطور(DPS). المجموعة الرابعة و الخامسة كلاهما قسيتا عند (760 ºC) ،لكن المجموعة الرابعة أخضعت إلى المعاملة الزمهريرية و المجموعة الخامسة أخضعت للمعاملة الزمهريرية ثم المراجعة عند (200 ºC) مدة ساعة واحدة. تم إجراء فحص الشد والصلادة علاوة على الفحص المجهري. ان مقاومة الخضوع والشد و كذلك المطيلية مرتبطة بنسبة المارتنسايت. مقاومة الخضوع والشد للصلب ثنائي الطور(DP) لأنواع الصلب الثلاثة أعلى من الصلب المطبع، و لوحظ ارتفاعها بعد المعاملة الزمهريرية، وان هذه القيم انخفضت بعد المراجعة عند (200 ºC). المراجعة عند درجة حرارة (200 ºC) للصلب الثنائي الطور (DP) بعد المعاملة الزمهريرية أدت إلى عودة نقطة الخضوع لنوعي الصلب (C12D, C32D) بينما لم يلاحظ هكذا تغيير بالنسبة للصلب (C42D). كذلك لوحظ ان الصلادة للصلب الثنائي الطورازدادت بعد المعاملة الزمهريرية ولأنواع الصلب الثلاثة.

**الكلماث الرئيسية: معاملة زمهريرية، صلب ثنائى الطور، خواص الشد، الصلادة، الصلب .**

## 1. INTRODUCTION

Prior to dual-Phase steel developments, it have been focused primarily on applications involving cold-or hot-rolled sheet containing about 0.1% C. Carbon content was kept low because of the need for good spot weldability recent developments have been directed at producing dual-phase steels with high carbon contents (0.2 to 0.4) for use in forging or bar applications (S. Tasuhara et al, 1987). Medium carbon low alloy steel grades are widely utilized for the design of components and structural parts for moderate to high stress applications. Such applications require the selection of materials having an optimum combination of high strength, ductility, and toughness for effective service performance. Moreover, failure of stress bearing components can be catastrophic in nature, leading to grave economic and technical losses (Soboyejo W 2003, Brobergs KB. 1999). It is for these reasons there is a sustained interest in the development of steel microstructures with excellent high strength and toughness characteristics (Lozano JA, 2008, Salehi AR et al., 2006). The development of high strength - ductile microstructures in medium carbon low alloy steel has been explored with the adoption of intercritical treatment with encouraging results obtained (Alaneme KK and Kamma CM 2010), (Alaneme KK et al., 2010) .The tensile and fatigue properties of dual phase microstructures produced in medium carbon low alloy steels by intercritical treatment have been reported to be superior to that obtained from conventional heat-treatment processes (Alaneme KK, 2010). Ferrite martensite dual phase (DP) steels have microstructure containing hard martensite islands embedded in a soft and ductile ferrite matrix. this unique microstructure result in characteristic mechanical properties like absence of yield point phenomena, large ratio of tensile strength to yield strength, high rates of work hardening, high total and uniform elongation, excellent forming characteristic and high fracture toughness (M.S. Rashid, 1981, S. Hayami, 1975) . It has, thus become possible to employ DP steel widely in automobile components such as bodies, chassis, bumpers, wheel discs and rims (M.S. Rashid, 1981).

Dual phase steels have been designed to have low carbon with or without alloying elements and heat treated or hot rolled to have martensite volume fractions (MVF) rarely exceeding 15% because beyond this percentage, formability of DP steels is b**adly affected (M.S. Rashid, 1981 ).** The ensemble of the stronger phases, martensite, bainite and pearlite, is often referred to as "second phase ", retained austenite is also generally present in DP steels due to uncompleted austenite to martensite transformation. The amount of retained austenite varies from 2 to 9 % with composition, tending to be more prevalent in steels with high carbon content and other alloying elements **(G.R. Speich, 1981)**.

A cryogenic treatment is the process of treating work pieces to cryogenic temperatures (i.e. below -190 ºC) to remove residual stresses and improve wear resistance on steel. The process has a wide range of applications from industrial tooling to improvement of musical signal transmission. Some of the processes benefits include longer part life, less failure due to cracking, reduce coefficient of friction an less electrical resistance. This kind of treatment apart from identical effects as in the event of traditional cold treatment is also accompanied by crystallographic and microstuctural changes thanks to which as a result of tempering process carried out after cryogenic treatment in the steel microstructure very fine carbide precipitations occur. These precipitations affect both the material strength and its wear resistance (Collins D.N., 1996, Huang J.Y., 2003). In the present work the (C12D, C32D &C42D) steels were intercritically heat treated and effect of martensite content on tensile properties and effect cryogenic treatment on its properties and microstructure was examined.

## 2. MATERIALS AND METHODS

### 2-1 Materials

The steels grades used in this research were selected from standard types of carbon steel, with different carbon content ranging from low to medium. This helps to understand the effect of carbon content and cryogenic treatment on the properties of dual phase steel. The tested steels, who is chemical compositions shown in table1, designated (C12D, C32DandC42D), according to the German standards specifications (DIN).

### 2-2Heat Treatments

The heat treatment included normalizing, intercritical annealing and tempering treatment as shown in Table 2. Intercritical annealing carried out at (760 ºC), and the holding time was (30 min), followed by quenching in Brine **solution**

(10% NaCl) to obtain (DP) steel. Tempering was carried at (200 ºC) for one hour, and was employed after cryogenic treatment. The research methodology is shown in Fig. 1.

## 2-3 Cryogenic Treatment

Liquid nitrogen was used as cooling media to carry out the cryogenic treatment. The specimens were encased in paraffin wax to act as insulator. This enables gradual change in the temperature of the specimen to prevent any thermal shock that may occur, thus causing undesirable deformation or cracking. When cryogenic treatment temperature (-196 ºC) is reached, the components then held at that temperature for a period of 24 hours, after that they were removed and left to warm at room temperature. The cryogenic treatment of the specimens was done in a chamber which is fully covered with multilayer super insulation and is filled by liquid nitrogen.

## 3-MECHANICAL PROPERTIES TESTS

## 3-1 Tensile Testing

The tests were performed using tensile testing machine type (United Hydraulic SHFM Series), in Specialized Institute for Engineering Industries\ Ministry of Industry. The test machine has a maximum capacity of (100KN) with a speed of 1 mm/min. Two specimens are used to perform each tensile test.

### 3-1-1 Specimen for Tensile Test

The tensile test specimens have been prepared in a accordance with the German standard for testing materials (DIN). They are of a cylindrical shape type, and are shown in fig. 2.

## 3-2 Microhardness Testing

In present work Vickers microhardness tester type (HVS-1000) was used, with pyramid indenter and load (500 g), in Ministry of Sciences & Technology. The specimens used for this test were cylindrical shape (ϕ15 mm x8 mm).

## 4- RESULTS AND DISCUSSION

## 4-1 Tensile Properties

The results of tensile propeties test i.e yield strength(σy), ultimate tensile strength(σu), and percentage elongation (El%) are presented in tables 3 and Fig. 3, 4 and 5, for (C12D), (C32D) and (C42D) steels respectively. The key factor which differentiates dual-phase steel from the variety of the ferritic steels (e.g. mild steels and conventional HSLA) with respect to the deformation characteristics, is the absence of yield point.The ferritic-martensitic steels exhibit continuous yielding. This feature together with the very high initial strain hardening rates account for the excellent formability properties of dual-phase steels. The continuous yielding of dual phase steel remains after cryogenic treatment ,but after tempering at temperature (200 ºC) for one hour, the yield point reappear for (C12D) and (C32D) steel, but it is less clear in the later steel. As for (C42D) steel, the tensile profile did not show such effect. For the three grades of steel , both tensile and yield strength (σu, σy) of dual phase steel are higher than those of normalized steel, this is due to the martensite presence in the structure of dual phase steel, whereas, normalized steel consist of the lower hardness pearlite. The improvement in tensile strength and yield strength follow a similar trend with increasing martensite content. These results are not surprising since martensite is a hard phase. As the martensite content increases, it is expected that the strength of dual phase steel increases but at the same time brings brittleness to the steel. But the benefit here, is the increase in the difference between (σu) and (σy) after (DP) treatment, because the greater the difference between the yield and ultimate tensile strength, and the further of the material can be stretched prior to necking (Richard Gendny, 2002).Table 4 represents these differences measured from tensile test profiles taking into account the approximate actual (σy) and not the 0.2% proof stress

The best documented cryogenic treatment mechanism affecting steel, other than causing the transformation austenite-to-martensit, is the precipitation of strengthening phases, in particular the carbide phase (Collins D. N., 1996).Therefore, the martensite content of dual phase steel has been increase after cryogenic treatment for this cycle and this result in both (σu) and (σy) increased after cryogenic treatment.

## 4-2 Microhardness Property

Microhardness tests after (DP) treatment. Shows an increase by (47.56%), (44.84%) and (9.02%) for the above steels respectively over those of normalized specimens. This increase in hardness is directly related to the nature of dual phase steel microstructure, which consist of a hard phase martensite and the soft phase ferrite, whereas normalized steel consist of a less hard pearlite and ferrite. Also some of the carbon and other element, which remains in solution of the ferrite phase after quenching, contributes to a small increase in hardness. The differences in hardness between the (DP) of these steels, are due to the volume fraction of martensite, which increases as the carbon content of the steel increased, hence it is logical to expect an increase in hardness of dual phase steel with increasing martensite volume fraction at the expense of the ferrite phase.

After cryogenic treatment, the hardness increased by (71.33%), (58.00%) and (13.25%) respectively over those of normalized condition or by (16.11%), (9.08%) and (3.88%) over those of (DP) condition. The response of dual-phase (C12D) and (C32D) steel to cryogenic treatment is clear and showed positive increase in hardness while (C42D) steel shows only moderate increase, which means that (DP) steel of a high martensite volume fraction due to higher carbon content, have moderate response to cryogenic treatment concerning hardness. This could lead to a conclusion that changes in the ferrite phase structure after cryogenic treatment have a major contribution to the increase in the hardness of (C12D) and (C32D) steel. The microstructure presented in Fig. 9 shows the ferrite phase after cryogenic treatment contains finely distributed precipitates.

## 4- MICROSTRUCTURE TEST

### 4-1 Microstructure of C12D Steel

The microstructure of (DP) steel, before and after cryogenic treatment is shown in Fig. 8and 9. By using point counting method, it was found that martensite content approximately (21%).For (DP) after cryogenic treatment and dual phase after (cryogenic + tempering ) treatment, it was found that martensite content for both conditions approximately (25%). This small increase in martensite content may attribute to the transformation of retained austenite to martensite after cryogenic treatment. Also it can be noticed that the ferrite in (DP) steel after cryogenic treatment contains finely distribution precipitates as shown in fig. 9.

### 4-2 Microstructure of (C32D) steel

Fig. 10, 11 are microstructure of (DP) steel before and after cryogenic treatment. The martensite content of dual phase steel is approximately (37%), and after cryogenic treatments, the amount of martensite was found to be the same. After tempering, the structure showing some changes in which the martensite areas contains fine precipitates and the boundaries between martinsite and the ferrite is no longer sharp.

### 4-3 Microstructure of (C42D) steel

Fig. 12, 13 are microstructure of (DP) steel before and after cryogenic treatment, The martensite content of dual phase steel is approximately (78%), and after cryogenic treatments, the amount of martensite was found to be the same. However after (cryogenic + tempering) treatment there are clear changes, the microstructure contain fine precipitates throughout the microstructure.

## 5- CONCLUSIONS

- Quenching from temperature of (760 ºC) (Intercritical Annealing) produces a mixture of ferrite and martensite, i.e. (dual phase steel). The volume fractions of martensite were 21%, 37% and 78% for (C12D, C32D & C42D) steels respectively, and cause continuous yielding (no yield point) for the three steel grades.
- Cryogenic treatment carried out on dual phase steels did not cause the reappearance of the yielding point.
- Tempering treatment at temperature (200 ºC) for one hour causes the reappearance of the yielding point for steels (C12D & C32D), while no such a change was noticed in (C42D) steel.

- 4-A further small increase in $\sigma y$ and $\sigma u$ can be achieved by cryogenic treatment.

- 5-The response of carbon dual phase steels to cryogenic treatment largely influenced by the carbon content. Lower carbon steels (C12D) and (C32D) generally has higher response than (C42D) of higher carbon content.

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**Table 1: Showing chemical composition of the investigated steels.**

| Steels<br>%<br>Elements | C12D | C32D | C42D |
|---|---|---|---|
| C | 0.12 | 0.33 | 0.43 |
| Si | 0.25 | 0.276 | 0.209 |
| Mn | 0.58 | 0.527 | 0.673 |
| P | 0.012 | 0.023 | 0.018 |
| S | 0.035 | 0.045 | 0.010 |
| Cr | 0.087 | 0.142 | 0.132 |
| Mo | 0.015 | 0.016 | 0.017 |
| Ni | 0.073 | 0.057 | 0.070 |
| Cu | 0.174 | 0.011 | 0.118 |

Table 2: Showing details of the heat treatments used in the present work.

| steel | Heat treatment | Temperature ( °C ) | Holding time (min) | Cooling medium |
|---|---|---|---|---|
| C12D | N | 900 | 30 | Air |
| | IA | 760 | 30 | Brine |
| | T | 200 | 60 | water |
| C32D | N | 860 | 30 | Air |
| | IQ | 760 | 30 | Brine |
| | T | 200 | 60 | water |
| C42D | N | 850 | 30 | Air |
| | IQ | 760 | 30 | Brine |
| | T | 200 | 60 | water |

Table 3: Results of the tensile tests .

| Steels | Parameters | Type of treatment | | | | |
|---|---|---|---|---|---|---|
| | | N | N+CT+T | DP | DP+CT | DP+CT+T |
| C12D | σy(MPa) | 355.28 | 367.84 | 467.71 | 479.31 | 435.52 |
| | σu(MPa) | 494.6 | 497.24 | 669.68 | 704.01 | 572.57 |
| | El% | 33.61 | 35.25 | 22.05 | 18.01 | 24.60 |
| C32D | σy(MPa) | 364.16 | 393.73 | 473.3 | 526.61 | 386.85 |
| | σu(MPa) | 591.13 | 589.66 | 692.74 | 736.64 | 594.67 |
| | El% | 27.03 | 29.45 | 19.87 | 17.12 | 23.58 |
| C42D | σy(MPa) | 488.48 | 488.6 | 551.75 | 563.62 | 525.57 |
| | σu(MPa) | 700.01 | 696.1 | 742.79 | 755.17 | 715.43 |
| | El% | 24.22 | 26.11 | 19.16 | 17.86 | 20.46 |

Table 4: The difference between σu and the actual σy for normalizing condition and (DP) of the tested steels.

| Steel grade | (σu-σy) MPa Normalized | (σu - σy) MPa Dual phase | Increase (MPa) |
|---|---|---|---|
| C12D | 139.32 | 273.68 | 134.36 |
| C32D | 226.97 | 271.74 | 44.77 |
| C42D | 211.53 | 240.79 | 29.26 |

| $d_o$ | $d_1$ | $L_O$ | $L_c$ | $L_t$ | h | R |
|---|---|---|---|---|---|---|
| 8 | 10 | 40 | 48 | 115 | 30 | 4 |

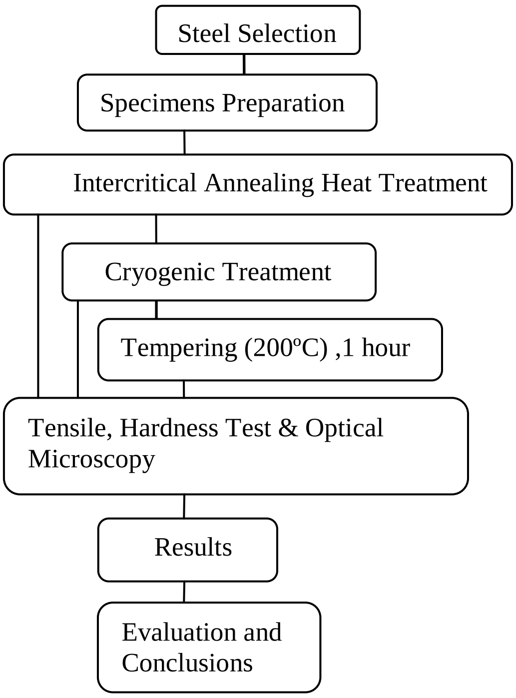

Fig. (1) Research Methodology

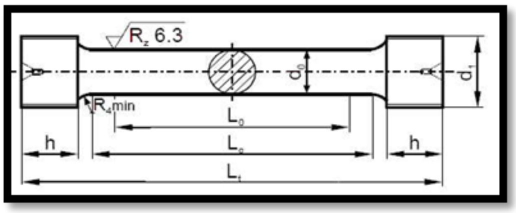

Fig. (2) Schematic of the tensile test specimen (all dimensions are in mm)

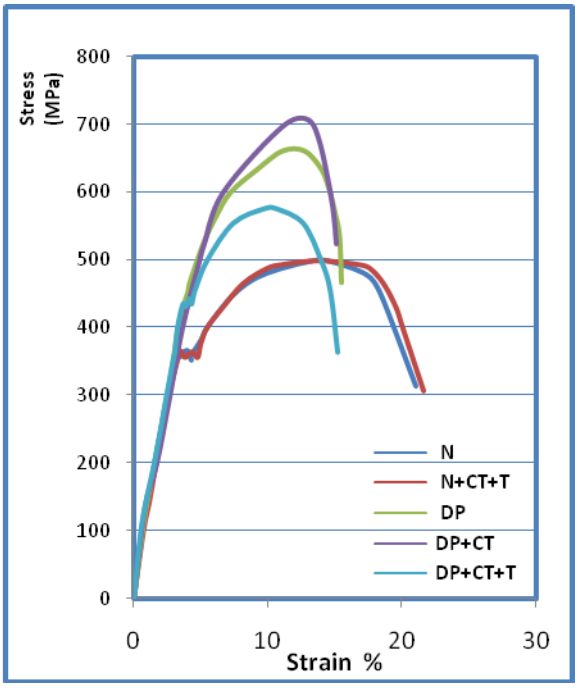

Fig. (3) Tensile test curves for C12D steel.

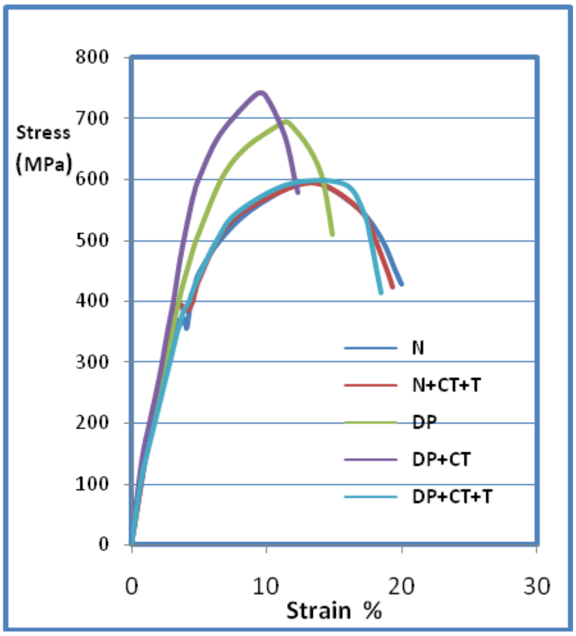

Fig. (4) Tensile test curves for (C32D) steel.

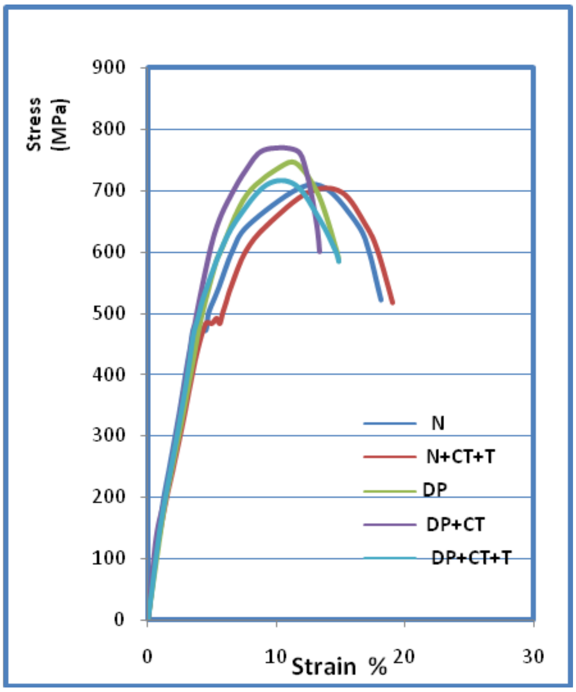

Fig. (5) Tensile test curves for C42D steel.

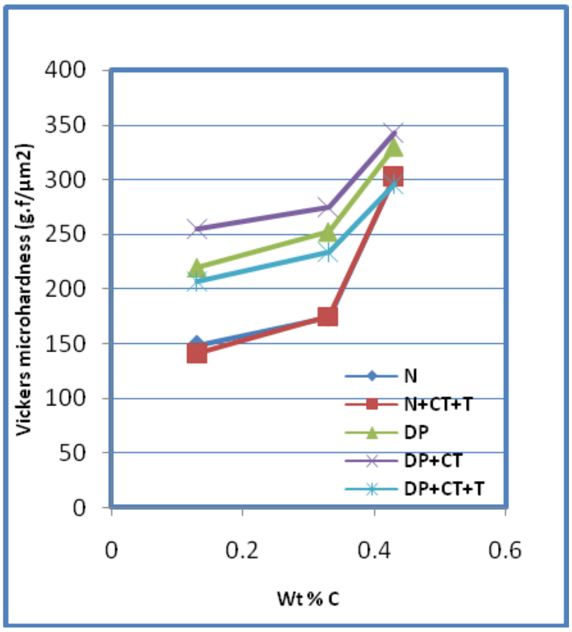

Fig. (7) Effect of carbon content on the response of dual phase steel to cryogenic treatment.

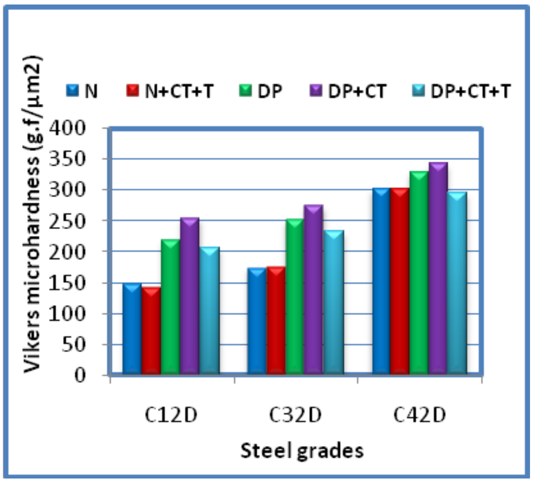

Fig.( 6) Hardness level and the response of each steel to cryogenic treatment.

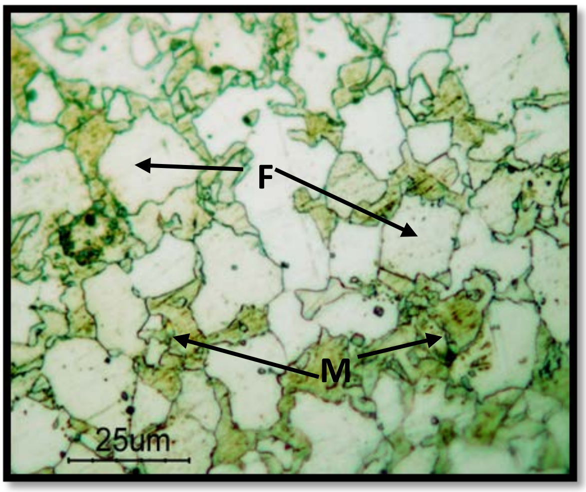

Fig. ( 8) Microstructure of dual phase for (C12D) steel. Ferrite (F), Martesit (M).

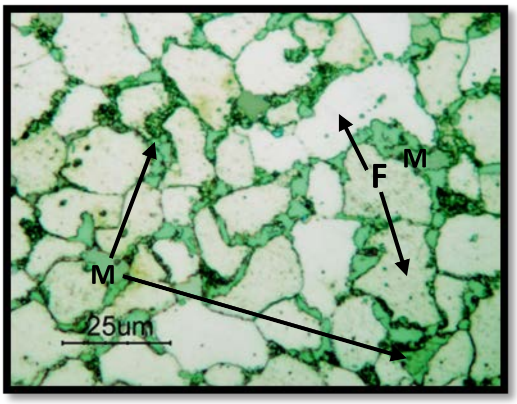

Fig.(9) Microstructure of dual phase after cryogenic treatment for (C12D) steel sowing fine precipitates within Ferrite grains.

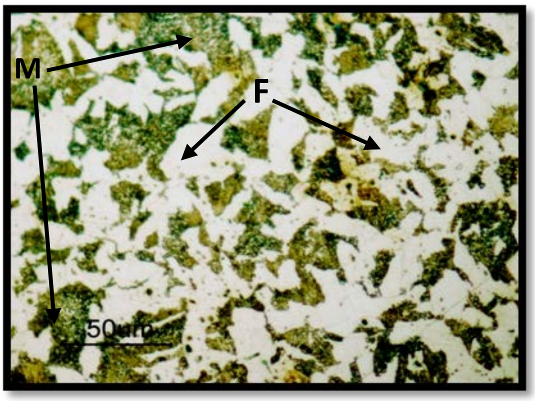

Fig. (10) Microstructure of dual phase for (C32D) steel. Ferrite(light), martesite(dark).

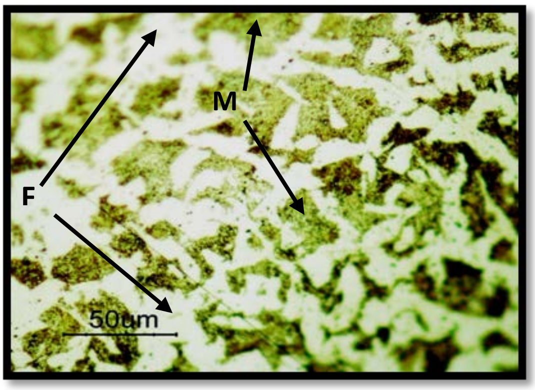

Fig. (11) Microstructure of dual phase after cryogenic treatment for (C32D) steel.

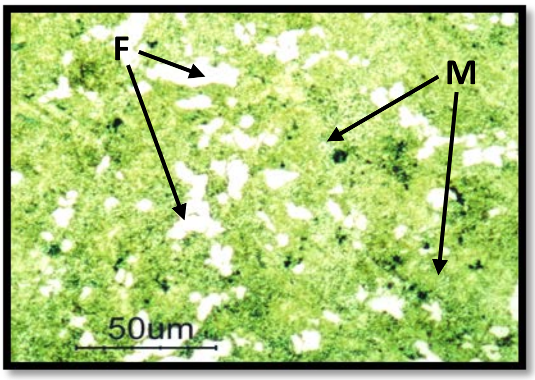

Fig.(12) Microstructure of dual phase (C42D) steel.

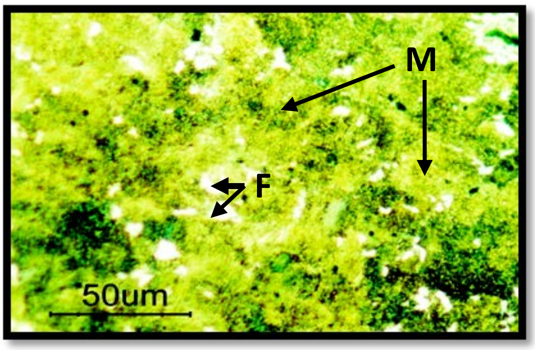

Fig. (13) Microstructure of dual phase (C42D) steel after cryogenic treatment.

| Abbreviation | The meaning |
|---|---|
| σy | Yield stress |
| σu | Tensile stress |
| DP steel | Dual phase steel |
| MVF | Martensite volume fraction |
| El% | Percentage elongation |
| N | Normalizing treatment |
| CT | Cryogenic treatment |
| T | Tempering treatment |
| 0.2 PF | Proof stress |
| IA | Intercritical annealing |
| HSLA | High strength low alloy steel |
| M | Martensite |
| F | Ferrite |