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RESEARCH ARTICLE

OBTAINING MULTIPHASE STRUCTURE WITH A SUBCRITICAL ANNEALING FOLLOWED BY QUENCHING IN WATER/OIL DIN CK55 STEEL

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ABSTRACT

Today, in the metallurgical industry there is a tendency to design new procedures or improve the classics, in the sense of an energy saving, reduction of processing steps and less environmental impact. With the process proposed in this paper, we try to replacing the classic thermal treatment for carbon steel in water / oil quenching and tempering, for one single stage subcritical annealing and quenching in water / oil; the tempering step is removed, and the mechanical properties obtained with the classical treatment are improved. The research was carried out with construction carbon steel (DIN CK55), manufactured by ThyssenKrupp Materials Ibérica, SA, achieving a complex multiphase structure of excellent mechanical performance.

INTRODUCTION

The importance of the steel industry is beyond doubt; being, in percent by volume, the material more used if we exclude the concrete. Even in the current construction, major building rules require a minimum equivalent of iron per cubic meter of concrete, especially in structures, clearly subjected to tensile, bending and compression, given the tenacity to bring the steel corrugated to design. Some steel production data for 2005 in millions of tons, are: Europe: 331; North and Central America: 134; South America: 45; Asia: 508; and the rest of the world: 39.3; appreciating significant growth in China and India with 24.6% and 16.7%, respectively, compared to 2004.

Researches in this field, at present, are aimed mainly at improving the effectiveness of the products designed, saving production time and minimizing environmental impact.

Chemical Composition

Table.1 Steel Chemical composition

Chemical Composition(%)	C	Mn	Si	P	S
Nominal	0.55	0.55	0.22		
Tolerances	0.50-0.60	0.40-0.70	0.15-0.30	0.04	0.04

In this research we propose a thermal treatment that optimizes the classic to improve resistance: the quenched and tempered by a more simplified treatment of subcritical annealing followed by quenching in water / oil. With this treatment we want to achieve the same mechanical strength, saving in time and processing technology, leading to an evident saving of energy and, therefore, less environmental impact.

For this we have selected a construction carbon steel according toDIN, the CK55 standard and its equivalent UNE ASTM F-1150 and 1055. (Table.1) (Stahlschlüssel, Verlag Stahalschlüssel Wegst Gm GH; 1992).

The limitations are its thickness, which should not exceed the critical diameter (Jominy) of quenching in oil / water = 11 / 7mm. (Stahlschlüssel, Stahalschlüssel Wegst GmgH Verlag, 1992).

Other major advantages of this steel, in addition to the above, improved toughness are obtained with the proposed treatment and the very competitive price of raw materials, coupled with savings in production costs.

The multiphase structure obtained with our proposed method provides some features that can be combined according to the

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percentages of phases present in the structure obtained (Guidelines, 2005; Matlock, 2011; Oliver *et al*, 2007; Delince *et al*, 2007; Frommeyer, 2003).

The improvement in toughness for a given resistance, safety binds to the type of fracture. Although there have been no fatigue tests, it is expected to improve the strength and fatigue life.

Experimental Technique

For this research were acquired CK55 steel bars, hot rolled and normalized, dimensions 240 mm long and 10 mm Ø section.

From these samples were prepared of different geometries, one of these to a heat treatment and other for tensile testing. The dimensions of the samples used in the heat treatment are 10 mm diameter and 7 mm long; while the dimensions of the samples subjected to tensile tests have dimensions of 12.6 mm initial section, 4 mm diameter and 19.7 mm in length, in line with the UNE 36011 and 111150 INTA that recommends samples of a maximum diameter of 11mm (Stahlschlüssel, Stahalschlüssel Wegst GmgH Verlag, 1992) (Figure.1).

Figure.1 cylinders samples made from the starting material.

The heat treatments were carried out in a muffle with thermal heating capacity 1100ºC, and these were (Figure.2):

Quenching: heating to 850 ° C, which is the austenitizing temperature for 25 minutes, with subsequent rapid cooling. Quenching were conducted both in water and in oil.

Annealing: heating to 850 ° C, which is the austenitizing temperature for 25 minutes, with subsequent slow cooling (oven).

Subcritical annealing: heating to 750 ° C below the critical temperature of a conventional annealing for 25 minutes, with subsequent rapid cooling. Quenching were conducted both in water and in oil.

To perform a correct observation metallographic the preparation of the samples was performed in a conventional manner: roughing in water with abrasive papers 120-240-600 and subsequent ly, polishing with alumina a (0.3µm) in water and polished finish with MasterPolish (0.05µm), all of Buehler. Chemical etching to reveal the structure of steel is achieved with reagent of Nital (4%) for 30 seconds, with subsequent washing in water and drying with ethanol in a stream of hot air. The techniques used for metallographic observation carried out in this research were conventional optical microscopy, with an optical microscope Reichert MEF4 A / M with digital camera Canon IXUS 575 7.1 megapixels built-it, and scanning electron microscopy with a JEOL JSM -6400, operated at 20keV, which incorporates a Oxiford Link 6506 system Pentafet energy dispersiveanalysis (EDX) X-rays and a backscattered electron detector (EDS).

Figure.2 Samples with thermical treatment.

To determine hardness Vickers was used a durometer Vickers AKASHI AVK-AII 50kg maximum load. The test was carried out, applying a load of 30 kg, with a time of penetration of 20 seconds.

For determination precise and clear of micrographic phases obtained during heat treatments, was used microdurometer Vickers AKASHI MUK-E3, which allows, localized, select the area to will be determined. The norm followed for lengths diagonal footprint between 0.020mm and 1.400mm has been the UNE-EN ISO 6507-1: 2006. The obtained data are an average of 15 measurements per test piece (Stahlschlüssel, Verlag Stahalschlüssel Wegst GmgH; 1992). Determining the Rockwell C hardness it was performed in a universal durometer Officine Galileo mod. A200, maximum load 185kg. The test was carried out, applying a load of 150kg with a penetration time of 20 seconds. The obtained data are an average of 5 measurements per test piece. In this case the norm has been followed the UNE-EN ISO 6508-1: 2007 (Stahlschlüssel, Stahalschlüssel Wegst GmgH Verlag, 1992). The tensile test was conducted on a SERVISIS ME10 KN with PCD2K operating system. The tensile test was carried out increasing the load with a controlled rate of 10 MPa / s, recording the values of load and deformation produced.

RESULTS

By optical and scanning electron microscopy it has been possible to observe the microstructure of the samples studied. Both techniques show very clear structures of the steel understudy DIN CK55. Thus, in the case of steel starting, this is a steel hot-worked and normalized, which is observed a laminar coarse pearlite structure and crystals of ferrite in grain austenitic boundaries.

Figure.3 micrograph by M.O. (X100), where the reception structure of the steel is observed, hotworked and normalized.

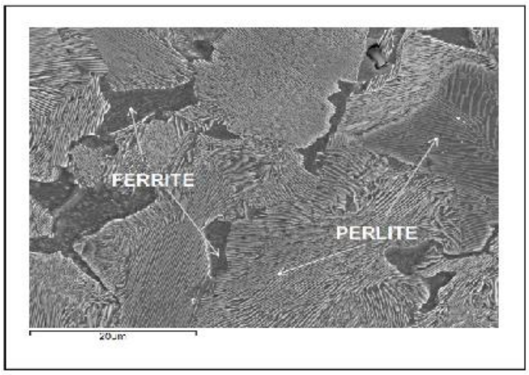

Figure.4 micrograph by SEM, where the reception structure of the steel is observed, hotworked and normalized.

In Figures 6 and 7, the DIN CK55 steel has been annealed at 850ºC for 25 minutes and quenching in water. It is seen, very clearly, acicular structure fully martensitic. For the thickness of the samples, the martensitic structure throughout the section.

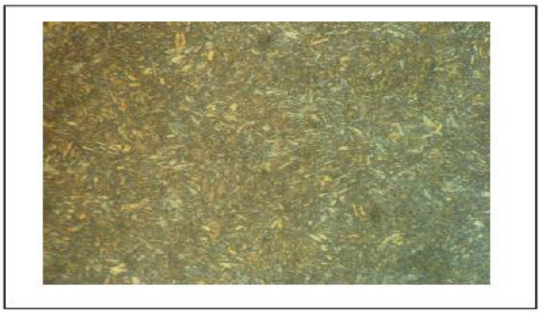

Figure.5 micrograph by M.O. (X50), in which the martensitic structure of a conventionally annealed steel is observed and water quenching.

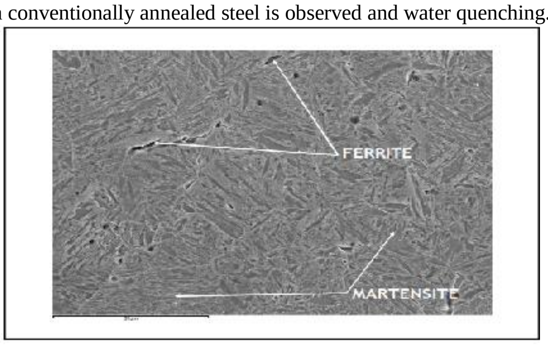

Figure.6micrograph by S.E.M., in which the martensitic structure of a conventionally annealed steel is observed and water quenching.

The same starting steel, DIN CK55, with subcritical annealing treatment at a temperature of 750 ° C for 25 minutes and water quenching; the microstructure is observed is complex, with lagoons of martensite, ferrite crystals, and colonies of pearlite, both fine and coarse (Figures 8 and 9).

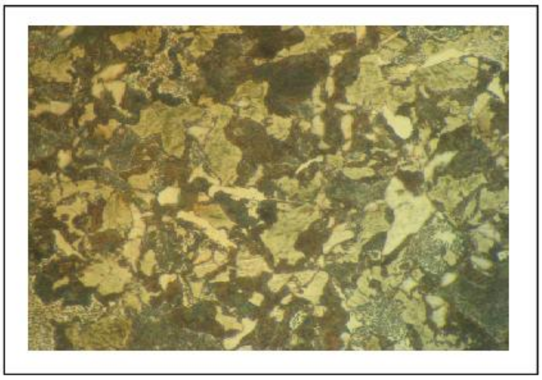

Figure.7 micrograph by M.O.(X50), in which a multiphase structure of the steel is observed DIN CK55, subcritical annealed at 750 ° C and water quenching.

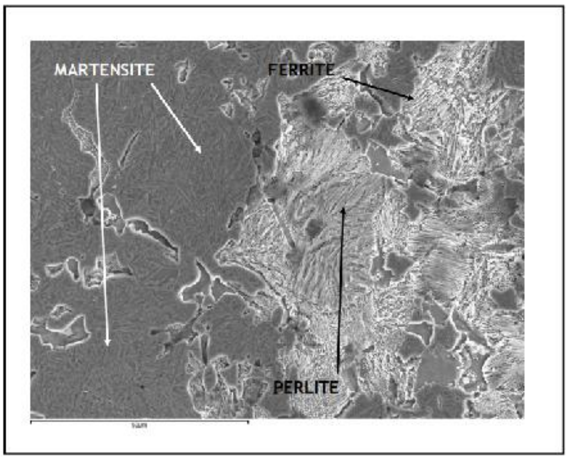

Figure.8 micrograph by SEM, in which a multiphase structure of the steel is observed DIN CK55, subcritical annealed at 750 ° C and water quenching.

The same steel, with the same subcritical annealing treatment, at a temperature of 750 ° C for 25 minutes, but oil quenching, appear the same micro constituents but different proportions: less marten site crystals and more proportion of fine perlite. This will significantly influence in lower hardness and strength, and increased toughness of the structure obtained (Figures 10 and 11).

Figure.9 micrograph by M.O. (X50), in which a multiphase structure of the steel is observed DIN CK55, subcritical annealed at 750 ° C and oil quenching.

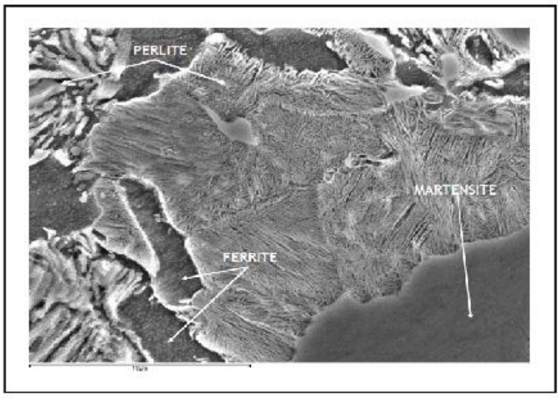

Figure.10 micrograph by S.E.M., in which a multiphase structure of the steel is observed DIN CK55, subcritical annealed at 750 ° C and oil quenching.

By Vickers hardness tests performed the results obtained were as follows (table.2):

Table.2 Vickers hardness values obtained are the result of 15 tests on each sample of steel, with different types of treatments.

Heat treatment	HVP
starting material, normalized and hot-worked.	**212**
water quenching and annealing convencional.	**698**
subcritical annealing quenching in water.	**498**
subcritical annealing quenching in oil.	**440**

By microhardness tests performed the results obtained were as follows (table.3):

Table.3 Microhardness values obtained are the result of 15 tests on each sample of steel, with different types of treatments.

Heat treatment	HVP
Starting material, normalized and hot-Worked.	180 (microconstituent pearlitic) 230 (ferritic crystals)
Water quenching and annealing convencional.	698
Subcritical annealing quenching in water.	152 (contour derrite crystals) 211 (contour coarse perlite) 344 (contour fine perlite) 620 (martensite contour)
Subcritical annealing quenching in oil.	140

By Rockwell-C hardness tests performed the results obtained were as follows (table.4):

Table.4 Rockwell-C hardness values obtained are the result of 15 tests on each sample of steel, with different types of treatments

Heat treatment	HRC
starting material, normalized and hot-worked.	16
water quenching and annealing convencional.	60-63
subcritical annealing quenching in water.	48
subcritical annealing quenching in oil.	41

By tensile tests performed the results obtained were as follows (table.5):

Below is a table of values (table.6) with the values obtained for DIN CK55 steelwith heat treatment and conventional values proposed for quenching and tempering classic.

Table.5 Tensile test values obtained are the result of 15 tests on each sample of steel, with different types of treatments.

Heat treatment	Mechanical Strength (MPa)	Elastic Limit (MPa)	Elongation (%)
starting material, normalized and hot- worked.	839.3	759.2	13 (ductile fracture)
subcritical annealing and quenching in water.	1113.6	930.5	4 (ductile- brittle fracture)
subcritical annealing and quenching in oil.	862.62	773.3	13 (ductile fracture)

Table.6 strength and elongation values of DIN CK55 steel with different heat treatments

	Mechanical strength (MPa)	Elastic limit (MPa)	Elongation (%)	Rockwell C Hardness (HRC)
A	839.3	759.2	13	16
B	1113.6	930.5	4	48
C	862.6	773.3	13	41
D	1150	1000	5	35
E	980	780	7	28
F	850	650	7	23
G	800	600	10	19

A: starting material, normalized and hot-worked.
B: subcritical annealing and quenching in water.
C: subcritical annealing and quenching in oil.
D: water quenching and annealing hard (*).
E: oil quenching and annealing hard (*).
F: water quenching and annealing tenacious (*).
G: oil quenching and annealing tenacious (*).
(* values collected from metallographic syllabus (Stahlschlüssel, Stahalschlüssel Wegst GmgH Verlag, 1992)).

DISCUSSION

With treatment of subcritical annealing and quenching in water/ oil, we have tried to obtain a DIN CK55 steel with the same or better mechanical characteristics with heat treatment that has been used today's industry, annealing at 850 ° C and quenching water / oil, subsequent, hard tempering at 450°C or tenacious at 650°C.

Clearly, the proposed treatment avoided the tempering step of(450-650)°C during (30-60) minutes, and the annealing step is lowered at the time and temperature, from 30 minutes at 850ºC to 15 minutes at 750 ° C. Is achieved, first delete a stage - tempering- which saves energy and time; and in second, energy is saved in the annealing step. This means shortening the processing time in the operation and saving energy consumption, while making an obvious economic savings, is clear an effect on the environmental impact. This, in principle, are very positive results to justify the new heat treatment; but it is justified if the mechanical properties obtained with the steel DIN CK55 are at least equal to or more positive than the conventional heat treatment.

Comparing the values obtained in our research with international metallurgical handbooks (Stahlschlüssel, Verlag Stahalschlüssel Wegst GmgH; 1992) can discuss the benefits of the mechanical properties of steel DIN CK55 undergoing treatment for subcritical quenching in water / oil.

Thus, with subcritical annealing and quenching water/oil, steel DIN CK55 gets, for annealing during 15 minutes, mechanical strength values similar to those obtained with conventional treatments. Values of 1113.6 and 862.6 MPa, for quenching in water / oil, are comparable data for 1150 and 850 MPa of strength, obtained by conventional annealing, quenching in water / oil and tempered. For values of elastic limit it occurs as with the mechanical strength. There is a great similarity.

Regarding the tenacity, that for a steel with similar resistance values, regardless of the heat treatment effected; this property can be gauged by the values of elongation (%). And these values confirm the objectives set for this research. Thus, for steel DIN CK55, under subcritical water quenching, the elongation value is 4%, compared to 5% of elongation, obtained by annealing, water quenching and tempering conventional hard, given by metalotécnicos handbooks (Stahlschlüssel, Stahalschlüssel Wegst GmgH Verlag, 1992) for this steel. The maximum advantage of heat treatment proposed in our research is when the temple is done in oil, comparable with conventional treatments, annealing, quenching in water / oil and tempered tenacious of metalotécnicos handbooks (Stahlschlüssel, Stahalschlüssel Wegst GmgH Verlag, 1992). In our case, the elongation achieved is 13%, compared to (7-10) % achieved with conventional treatments. Therefore, tenacity achieved with our treatment is higher than that achieved with the tenacious tempering after quenching in oil, described in handbooks. These elongations are obtained at equal values of mechanical strength and elastic limit. Our treated steel has therefore best security settings to use in any application.

Furthermore, it is known that the hardness values are related to the wear resistance: the higher hardness values greater resistance to this effect. Well, if we compare the data of HRC hardness obtained by steel DIN CK55, under subcritical quenching in water / oil with the same steel subjected to tempering and conventional tempered, they are frankly very favorable to our treatment of subcritical quenching, and hardness values of between 48 and 41 HRC, depending on whether quenched in water or oil are obtained, and from 35 to 20 HRC, if the conventional water or oil quenching is performed and applies a tough or tenacious tempering.

All these mechanical properties guarantee the goodness of subcritical annealing treatment and quenching in water / oil. If we add this to the advantages of reduced operating time, cut energy costs and reduced environmental impact, we think it would be very interesting to have this alternative heat treatment. We must emphasize that this treatment is truly effective and is proven to pieces not exceeding 9/11 millimeters thick advised by the Jominy test. They can be manufactured pieces of desired dimensions but this thickness limit; if these thicknesses are exceeded, subcritical annealing time would besuperior to homogenize the temperature throughout the room and would not beachieved in some areas, the tempering temperature subcritical water / oil.

CONCLUSIONS

This research try to replace the process of annealing, quenching and tempering conventional by other heat treatment shorter in operating time, saving manufacturing costs and minimizing environmental impact.

For this purpose, we have chosen the DIN CK55 steel, high carbon, used by construction, which has been treated to heat treatment subcritical annealing, short time, and quenching in water / oil. The limitations in the case of this steel, is the Jominy thickness, limiting its use to all kinds of sizes and shapes, but which critical thickness or diameter of less than 11 / 7mm to water / oil.

The maximum strengths achieved are all similar to those obtained by the conventional heat treatment. Subcritical annealed for 15 minutes and quenching in water / oil, resistance values obtained are 1113.6 and 862.6 MPa, respectively. The same applies to the limit of elasticity.

From elongation values obtained it is seen that for the subcritical quenching in water and the high resistance values, the data are consistent with the conventional heat treatment by water quenching and tempering hard. For the subcritical quenching in oil, the elongation is 13%, significantly higher than those obtained with treatment quenching in water / oil and conventional, hard and stubborn temper, ranging from 5% to 10%. From these data it follows that the parts obtained with subcritical quenching in oil, have a tenacity significantly higher than that associated with conventional heat treatments of quenching and tempering.

It follows from the HRC hardness values obtained from the samples subjected to subcritical quenching in water / oil, ranging from 41HRC to 48 HRC, faced with obtained in conventional thermal treatments, ranging from 35 HRC to 63 HRC, that the wear resistance is favorable to heat treatment proposed. In equal resistance, higher tenacity and wear resistance with subcritical quenching, faced with conventional quenching.

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