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Comparative analysis of microstructure and hardness of Isobloc W360 steel with tempering and Austempering heat treatment

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Introduction

In the Ecuadorian industry, the sector focused on taps and fittings is linked to the elaboration of dies for creating new brass elements or pieces, through the forging process that allows the production of a great number of equal products. This process presents as an advantage good mechanical property such as resistance to corrosion, fatigue, great tenacity and ductility (Quitiaquez et al., 2022). Due to the properties obtained by this process in the manufacturing field, forging accounts for 20-30 % of the value of all goods and products produced (Moreno & Adames, 2022).

The materials used in forging dies involve aspects that must be taken into account, such as the plastic deformation obtained by cold forging, to increase the ductility of brass, forging is carried out in hot forging (Penghui et al., 2020), Zuno et al. (Zuno-Silva et al., 2023) detail for hot forging works an addition of vanadium plus boron to be potentially used in dies obtaining an improvement in wear resistance, which translates into an extension in the useful life of forging dies. The addition of vanadium and boron produced a stable wear factor at a temperature of 22 and 400 °C.

When mentioning hot forging, factors such as working temperatures of approximately 1050 °C, are contemplated as the problems presented by forging dies. These problems are wear, mechanical fatigue, breakage, plastic deformation, and cracks due to thermal fatigue (Fernádez-Tamayo et al., 2022), and can be found in different parts of the cavity of the dies, thus dominating different failure mechanisms. Peñaloza (Peñalosa, 2022) details that the metal is heated to high temperatures before being worked to reduce its resistance to deformation. In the case of steel (depending on the alloy and carbon content), the temperature ranges between 800 and 1250 °C, being higher than the recrystallization and phase transition temperature, facilitating the forming of the alloy and the working of the dies in forging.

Tchiquendja et al. (Tchiquendja-Eleno et al., 2020) detail that the material used for tooling usually has 0.47 % carbon, 0.70 % manganese, and other alloys that should contain the steels for forging that are treated at high temperatures and for determining the appropriate type of cooling, obtaining an improvement in the mechanical properties to be used in hot forging processes. It is observed by microstructural analysis if the material improves its deformation strength or changes its structure. Ishtiaq et al. (Ishtiaq et al., 2022) mention that when tempering steel with a carbon content ranging between 0.37 and 0.54 %, it is evident in its microstructure the presence of martensite, which determines that the material acquires high hardness but increases its brittleness, thus being dismissed in hot forging works due to the presence of cracks.

Chien et al. (Huang et al., 2023) detail that not only does the amount of carbon interfere when a material is exposed to a heat treatment such as quenching. Described for 631 stainless steel, the working temperature in quenching is around 500 °C; treated with austempering at 720 °C, it is possible to obtain a higher tensile and impact strength than 631 stainless steel under normal quenching conditions. Properties are improved by the influence by the chemical composition, in this case, the amount of chromium (Cr) which is 16.99 %. Sabzalipour and Rashidi (Sabzalipour & Rashidi, 2023) performed the heat treatment of tempering and austempering, and the variation of hardness goes from 50 % of the hard phase of ferrite and martensite to different from those composed of ferrite-bainite phases, which goes from 80 % in volume of hard phase.

Lee et al. (2022) discuss nanostructured high-carbon bainitic steels austempered at 200, 250 and 300 °C. It is observed that all steels have mostly plate-like transformation products such as bainite. Plate-type bainite may contain retained austenite and martensite transformed from metastable austenite after quenching, especially in the steels austempered at 200 and 250 °C, which means that the austempered steels have a higher martensite fraction compared to the steel austempered at 300 °C. Brown et al. (2023) detail that bainitic steels exhibit strain hardening by improving the properties of ultimate tensile strength and hardness 600 to 670 HV compared to quenched and tempered steels.

Su et al. (2022; 2023) describe that for an M50 steel, a heat treatment of quenching plus three times tempering is applied, in which the solution temperature ranges between 1070 and 1110 °C and the tempering temperature is between 540 and 550 °C. After heat treatment, the microstructure of M50 steel consists mainly of quenched martensite and a small amount of retained austenite, hardness at room temperature can reach 60-63 HRC. The processes result in multiscale microstructure refinement that can improve the yield strength of martensitic steels (Wang et al., 2023). When considering the limits of a martensite microstructure, it is observed that an austempering contributes to improving the mechanical properties of the steels, García et al. 2022) detail the austempering heat treatment on a 0.41 % carbon (C) steel, which starts with an austenitizing at 950 °C for 72 min, followed by a molten salt bath at 310 °C for 120 min and then air-cooled. The salts used were a mixture of 45 % NaNO3 and 55 % KNO3. It has characteristics such as resistance, toughness, wear resistance, corrosion resistance, and shock absorption (Zhou et al., 2020).

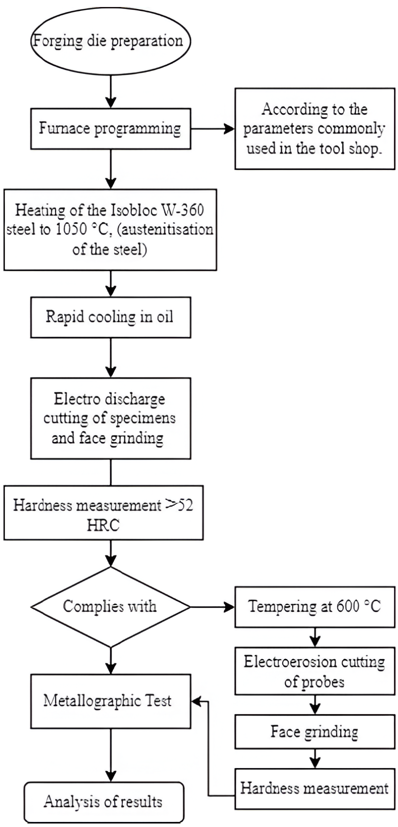
Behzadifar et al. (describe that an increase in the austempering time increases the carbon content of the retained austenite, regardless of the temperature and the microstructure obtained, which consists of bainitic ferrite plates with an average thickness of 53 nm surrounded by carbon-rich retained austenite with a hardness of 561HV, yield strength of 1614 MPa and a total elongation of 9 %. Varshney et al. (2020) similarly mention that the amount and size of carbides in the samples austempered at 350 and 400 °C are smaller than in the samples austempered at 300 °C.

This research aims to carry out a comparative analysis of the heat treatment quenching-reviving with tempering-reviving, to determine the treatment that avoids the presence of cracks in dies used in forging that is used in taps and fittings. The introduction describes the importance of heat treatment in stainless steel and mechanical properties that are improved with the heat treatments performed. The materials and methods section presents the heat treatments performed, the microstructure obtained, as well as the resulting hardness in each case. In the results section, the comparative analysis between the two treatments is detailed, analyzing the microstructure and the hardness obtained, thus determining which heat treatment is the most suitable to avoid the presence of cracks in the material when it is subjected to forging work.

Materials and Methods

In the heat treatment process, the chemical composition of the material and the temperatures recommended by the manufacturer for the heat treatments to be carried out must be clearly known, in addition to the Temperature, Time, Austen-ite Transformation (TTT) diagrams to determine the correct time and temperature for the experiment, as well as the microstructure obtained. Figure 1 indicates the procedure to be followed to carry out this research.

Figure 1
Procedure for evaluating the quenching and tempering process of Isobloc W-360



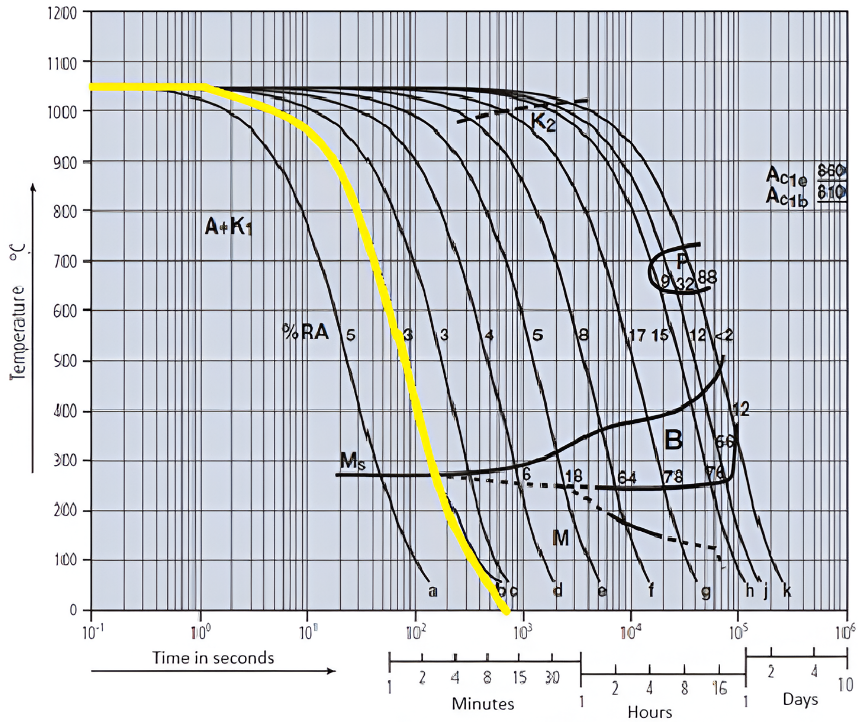
The Isobloc W-360 steel samples with dimensions of length, width and thickness of 50, 20 and 10 mm respectively, were the result of cutting with wire, avoiding that the material would modify its microstructure by the preheating of the cut. The chemical composition of the steel is detailed in table 1,where it is observed that it is a material that possesses a high content of Chromium as of Molybdenum can be exposed to works of high temperatures (Rodríguez-Cabrera, Ruíz Mondragón, Macías-López, 2019).

Table 1
Chemical composition of Isobloc W360 steel (%wt) (Bohler, 2021)

Carbon (C)	Silicon (Si)	Manganese (Mn)	Chrome (Cr)	Molybdenum (Mo)	Vanadium (V)
0.5	0.2	0.25	4.5	3	0.6

The processes for austempering, which is a heat treatment in which the surface of the low carbon steel is carburized and when a sudden cooling is applied, is subjected to an isothermal cycle, favoring the formation of a bainite base in the decarburized layer transforming it into a solid solution of quenching and austempering (Ríos-Diez et al., 2020). For the austempering, the curve 'S' of the TTT diagram corresponding to the Isobloc W-360 steel is used, from which the temperature and time for the phase transformation to Bainite is obtained, which is 300 °C and 30 min respectively, as shown in Figure 2. Curve ‘b’ is the one that resembles the cooling carried out in thermal oil.

Figure 2
Continuous quenching TTT curves of Isobloc W-360 steel (Bohler, 2021)



The process carried out for the quenching heat treatment starts with the furnace at a temperature of 650 °C heating for a set time of 35 min, generating nucleation and growth of acicular ferrite. Once it was kept in that time interval to reach austenitisation 1050 °C is applied on the samples without removing from the furnace, maintaining the time limit of 35 min continuously. When the determined temperature and time intervals are met, a rapid and uniform cooling in thermal oil is carried out until the fluid temperature is reached. Finally, the specimens are removed from the oil tank, leaving the specimens to reach room temperature. As seen in Figure 3, a temperature vs. time graph is shown, considering time cumulatively.

Figure 3
Flow diagrams for tempering heat treatment

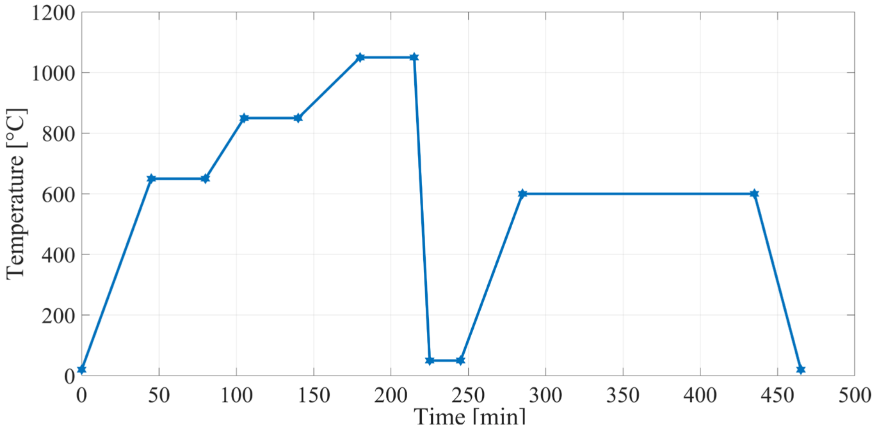
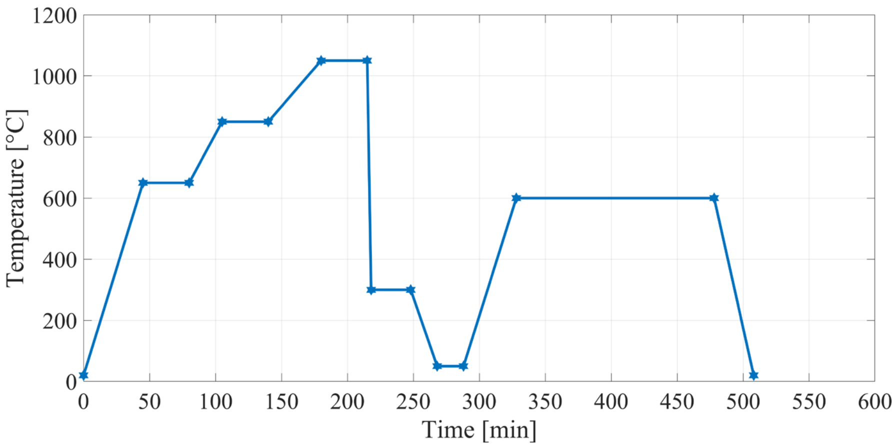


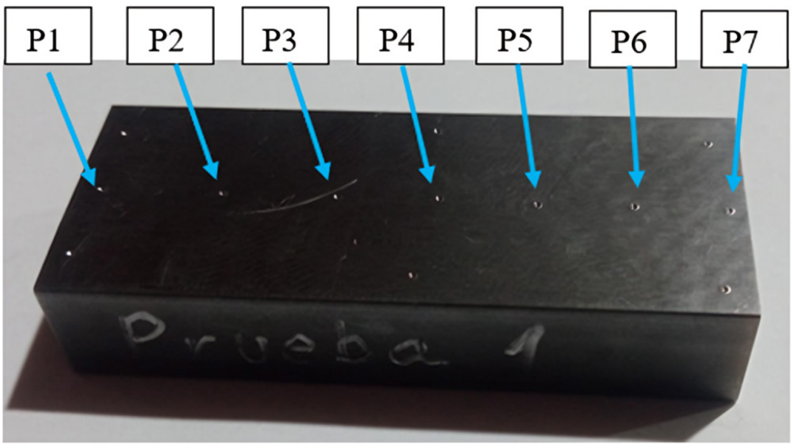
Figure 4 shows the temperature vs. time curve for the austempering treatment where the specimens are heated to a temperature of 650 °C and held for 35 min, continuing with an increase of 850 °C at the same time range where the temperature continues to increase to 1050 °C for 35 min, thus obtaining austenitisation. Unlike quenching, to obtain austempering, the samples are cooled in oil for 3 minutes so that they immediately reach a temperature of 300 °C, the samples enter the furnace which is preheated to 300 °C and is maintained for 30 minutes. Once this time has elapsed, they are cooled until they reach room temperature.

Figure 4
Flow diagrams for Austempering



As it is true that by performing only the heat treatment of quenching or austempering, an increase in the hardness of the material is obtained but this modification affects its brittleness and to prevent this from happening, a stabilisation tempering must be performed which should consist of a temperature of 600 °C and keep the specimens for 2 hours and 30 min. Leading to the Isobloc W-360 steel to balance its hardness as well as the microstructure to avoid causing brittleness in the material. Continuing with the analysis and to detail the existence of changes in the material we proceed to measure the hardness of the samples in the different stages of heat treatment, such as supply, quenching, austempering and tempering. In each of the cases, the measurements are taken in Rockwell (digital hardness tester TINIUS OLSEN FH 1-5) on HRC scale according to ISO 6508 or ASTM E18 (Require- et al., 2014). Five specimens were used for these measurements. Figure 5 shows the arrangement of the 7 points where the hardness measurements were taken.

Figure 5
Diagram of hardness measurements on the specimens



Once the hardness of the specimens that have been cut by wire EDM, the faces of the specimens are ground to obtain a uniform surface with the help of a flat grinder, obtaining a suitable surface finish to proceed with the mirror polishing of the specimens. The polishing of the samples is carried out with fine grain sandpaper under a water jet, and then followed by polishing with a cloth to obtain a mirror finish. Once each of the samples have these finishings, the chemical is applied to each of the test specimens. Nital_4 is applied for 11 minutes for the specimens that are only tempered and austempered, unlike the specimens that are tempered, Nital_4 is applied for 3 minutes and then prical for 40 seconds to obtain a clearer visibility of the microstructure of the material.

The qualitative metallographic analysis is performed with a NIKON TS2-S-SM optical microscope. It is a microscope designed to observe the microstructure of a material after being exposed to all the steps involved in a metallographic analysis. It is an inverted microscope with high resolution objective lenses 10X, 20X, 50X and 100X with very short working distances. To analyze the results, the 100X magnification lens is used to clearly appreciate the microstructures obtained by the thermal treatments applied to the specimens tested.

Results

Microstructure

As a first parameter of analysis, the microstructure is observed in each case of heat treatment in Figure 6. The microstructure of the tempered specimens is shown, which are constituted by a microstructure with a retained austenite base that are white areas that generate the visibility of the grain size. The presence of carbides dispersed in an intergranular way is observed leading the material to a phase where its hardness increases and the martensite sheets in black colour that like the carbides provide an increase in the hardness, but it goes hand in hand with the fragility generated in the material. In such a way that a stabilisation tempering is carried out in each of the cases.

Figure 6
Micrograph obtained after hardening

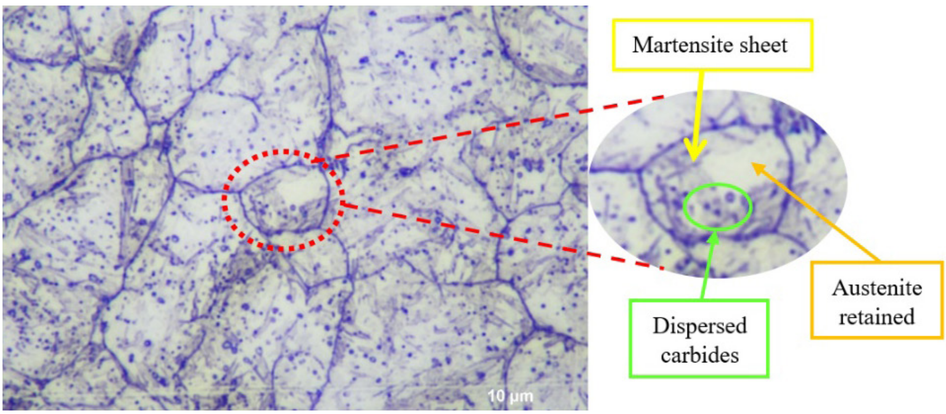
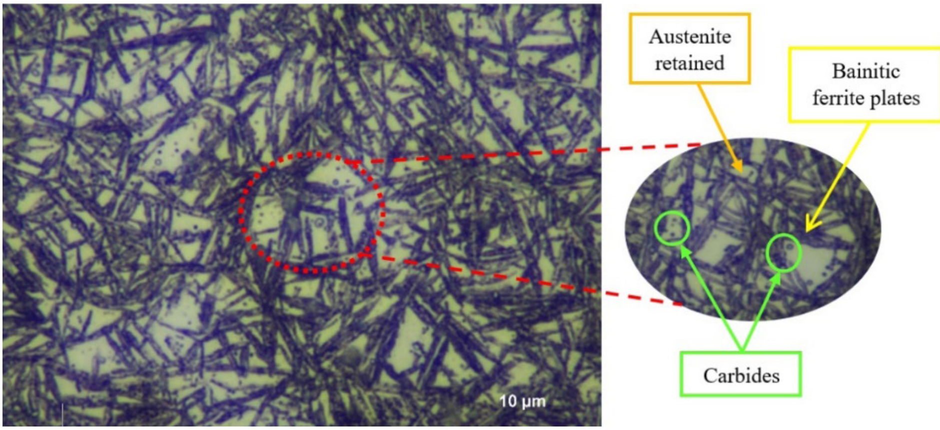


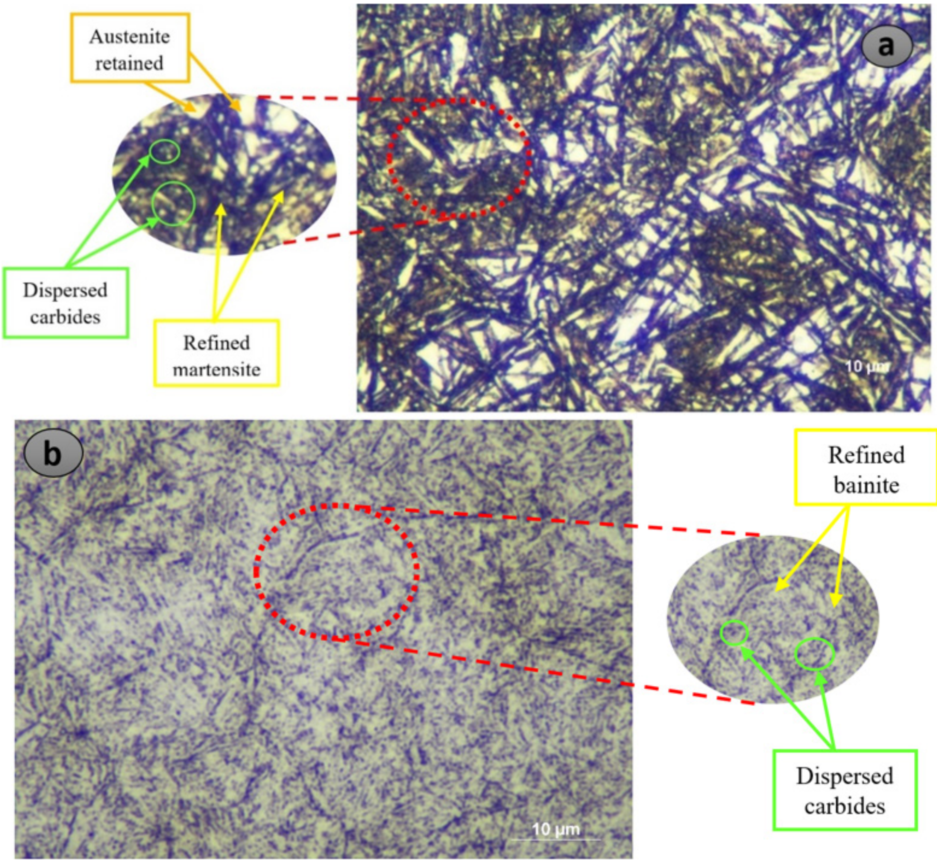
Figure 7. shows the microstructure of the austempered specimens, where it is clearly observed that the combination of intercritical austenitisation followed by the application of low temperatures (300 °C) generates the formation of bainitic ferrite sheets, which is more stable than the presence of austenite blocks, even so, the presence of retained austenite is evident, causing the release of nano-sized carbides which are formed by diffusion towards the austenite.

Figure 7
Micrograph obtained after austempering



As seen in Figure 8 in the specimens that were applied a stabilisation tempering for both tempered and austempered samples, in each case the tempering contributes with a stabilisation to the material. achieving structural homogeneity. In Figure 8 (a) a tempering can be seen after tempering, where the martensite sheets are refined by the plastic deformation of the martensite, generating a partial dissolution of carbide particles. The presence of dislocations and formation of carbon clusters contributes to the presence of nucleation of fine carbides during a subsequent tempering, as it is true that the influential factors for this phenomenon are temperature and residence time, but observing the presence of austenite retained as a base microstructure determines that the material improves the hardness. The low temperature in the shrinkage of the structure generates that the carbon atoms that are dispersed in the martensite and retained austenite jump to neighbouring dislocations and act as sites for the formation of carbides. Also, during tempering the high presence of Cr, Mo and W generate the significant presence of retained austenite as observed in the microstructure (Miranda, 2020). Figure 8 (b) corresponds to the application of tempering after austempering, and it is observed in the first phase that there is the presence of bainitic ferrite that when passing to a tempering of 600 °C, leaving a dwell time of 2 hours and 30 min, leading the cementite to precipitate into the bainite, thus generating ferrite plates which proceed to form segments parallel to the refined bainite. The form of precipitation of the carbides influences the stresses associated with the dysfunctional growth of the bainite, since the carbides in the bainite are extremely thin. As a consequence, bainitic ferrite tends to be more tenacious than refined bainite, despite being tougher, the sharp cementite particles of refined bainite act as nucleation points for crack dissociations (Ríos-Diez et al., 2020).

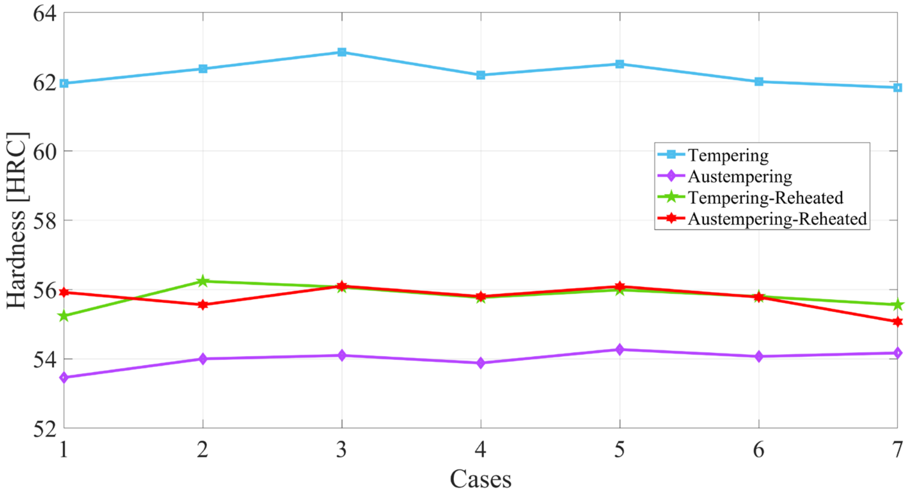
Figure 8
Micrograph obtained after tempering (a) quenched, (b) austempered



When observing the microstructure, the bainite is the result of an isothermal transformation. The size of bainitic ferrite plates and retained austenite is based on the application temperature, achieving considerable stability in the material when applying a heat treatment. Based on the study of the microstructure of the treated material, another parameter of analysis is observed. As for the hardness obtained from the samples after each thermal process, it is modified according to the time of permanence in conjunction with the exposure temperature. The supply hardness ranges from 5 to 7. 5 Rockwell hardness (HRC), when carrying out respectively the thermal treatment of tempering and austempering. It is observed that the hardness acquires the values of 62 HRC in the samples that are exposed to tempering, unlike the austempering specimens which decrease the hardness in a 12 % giving as value 54 HRC (Figure 9.), although it is true that the hardness is increased but the fragility of the material is high for forging works; therefore, the application of a stabilization tempering leads the material to present a homogeneous microstructure that goes hand in hand with a hardness of 56 HRC, achieving that the mechanical properties of the material can withstand high temperatures, avoiding the presence of cracks.

Figure 9

Micrograph obtained after tempering (a) quenched, (b) austempered



Conclusions

It is important to highlight that when heat treatments such as quenching and tempering are carried out, each one with a stabilization tempering in steels such as M50, W303, Isobloc W-350 of low carbon content, these contribute to carbide dispersions, but by containing alloys such as chromium and molybdenum these contribute to a uniform distribution of retained austenite as the base microstructure of the heat treatment of quenching and tempering, as well as in austempering and tempering. The presence of these alloys contributes to a homogeneous distribution of retained bainite, generating a significant improvement in the toughness of the material.

When a heat treatment is performed, the microstructure is obtained according to the amount of temperature and time exposed in the sample.A a change is generated in the Isobloc W-360 steel, when a tempering is performed. The presence of martensite as a base microstructure causes a hardness of 62 HRC, generating greater tensile strength but decreasing its ductility. Unlike only being exposed to an austempering with a hardness of 53 HRC obtaining a bainitic ferrite base microstructure as well as maintaining the mechanical properties of the material, when performing a stabilization tempering for both tempering and austempering it is evident that the hardness is matched both in the tempering-retempering and austempering-retempering, obtaining a hardness of 56 HRC; this value enters the range for hot forging work.

When performing the stabilization tempering in the two cases, it is evident that there is a decrease in hardness in the tempered-reduced of 9.5 %, which goes hand in hand with a microstructure of refined martensite base in conjunction with retained austenite, thus generating a stabilization of the mechanical properties improving its ductility. Unlike the austempering-reduced one, it presents an increase in hardness of 3. 6 %, with a microstructure of refined bainite and dispersion of carbides, thus stabilizing the mechanical properties and avoiding the fragility of the material, achieving in both cases an admissible hardness but with a microstructure in austempering-reduced avoiding a fragile structure, due to the temperatures used for the thermal treatments which must be established according to the properties to be improved.

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