Conjecturas

DOI: 10.53660/CONJ-934-K05

Procedures for preparing metallic specimens for submission to optical microscopy

Procedimentos de preparação de amostras metálicas para submissão à microscopia óptica

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ABSTRACT

Microscopy analyzes are very important for metallography, they serve to reveal the microstructural constituents, the distribution of present phases and other important properties to characterize the materials. It is important that the specimens follow a rigorous preparation process to ensure that they are free from defects that could compromise the results of the characterization. The work presents the stages of preparation of specimens for optical microscopy. Works related to the research proposal were analyzed in literature texts and thus it was possible to identify the most common and effective practices in the preparation phase of metallic samples.

Palavras-chave: Metallic specimen preparation; Microscopy; Preparation techniques for microscopy.

RESUMO

A microscopia é muito importante para a metalografia, pois serve para revelar os constituintes microestruturais, a distribuição das fases presentes e outras propriedades importantes para caracterizar os materiais. É importante que os corpos de prova sigam um rigoroso processo de preparação para garantir que estejam livres de defeitos que possam comprometer os resultados da caracterização. O trabalho apresenta as etapas de preparação de corpos de prova para microscopia óptica. Trabalhos relacionados à proposta de pesquisa foram analisados em textos da literatura e assim foi possível identificar as práticas mais comuns e eficazes na fase de preparação de amostras metálicas.

Keywords: Preparação de amostras metálicas; Microscopia; Técnicas de preparação para microscopia

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INTRODUCTION

Metallography relates the intrinsic structure of the material to its physical properties, processing steps, manufacturing to which they were subjected, and ultimately, the efficiency of their performance (ROHDE, 2010). In this sense, this examination makes it possible to reveal in a two-dimensional plane of intersection of the material's geometry, the constituents of microstructure, grain size, distribution of present phases and other characteristics that cannot be observed with the naked eye (GOKHALE, 2018; WARREN et al., 2016). Thus, optical and electronic microscopes appear as instruments used to enlarge the surfaces to be observed with the possibility of resolutions in the range of $10^{-3}$to 10^{-7}m (CALLISTER; RETHVISCH, 2016).

Therefore, it is extremely important to emphasize that the process of preparing the sample surface for submission to metallography must not present imperfections, scratches or defects that negatively influence the micrographic analysis, in a way that makes the analysis difficult or impossible, or even compromises the material to be observed (MALISKA, 2003; VOORT, 1987).

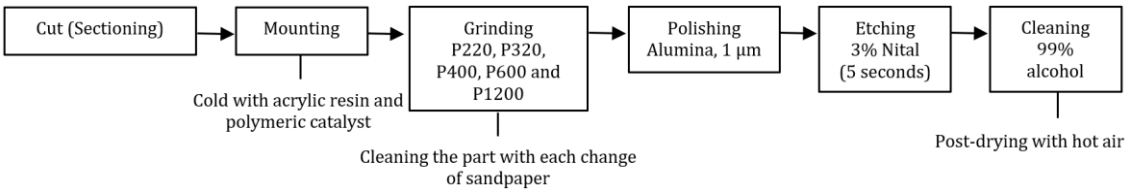
Given the above, the main objective of this work is to present a sample preparation methodology that offers a process flow with guaranteed reproducibility and standardization in microscopy and microhardness analyses. Furthermore, it is intended to list the main techniques commonly used in the literature and present them clearly, in a way that synthesizes each stage of preparation.

MATERIALS AND METHODS

In short, for the preparation of this article, a literature review was carried out focusing on research related to the preparation of metallic specimens for metallography (ABNT, 1995; ASTM INTERNATIONAL, 2011; COLPAERT, 2008; GEELS et al., 2007; ROHDE, 2010; ZIPPERIAN, 2009), with a focus on recent articles from databases: *Scopus*, *Science Direct*, *Ebesco*, *Google Scholar*, *Spring*, *ASM* e *Periódicos da Capes*. With this, starting with the identification of the most common sample preparation processes, the most pertinent parameters of each technique will be presented. To this end, the preparation sequence is illustrated in Figure 1 in which it was prepared in accordance

with the ASTM-E3:11 standard (ASTM INTERNATIONAL, 2011) given some adaptations and implementation of steps addressed in other bibliographies.

Figure 1 - Sample preparation sequence



Source: collection of the authors.

A. Cut

The cutting step is necessary when it is not possible to extract the samples in the appropriate dimensions for carrying out the tests and analysis of the material. With this, there are several cutting processes that can be commonly used for sectioning, among them, there are: shear; abrasive wear and fracture (MALISKA, 2003). It is worth mentioning that the cut should not influence the properties of the material, being indicated the removal of defective layers, deformed or that present flaws arising from the cutting process. However, microstructural changes may also occur as a result of frictional overheating at high speeds, this effect can be avoided by the correct use of coolant during cutting for procedures with abrasive wear (NORTON, 2013).

Furthermore, the shear cutting techniques commonly used and addressed in this step are indicated for low hardness and thin metals. It is, therefore, a fast process in relation to the others, which, in turn, generates excessive deformation and the emergence of residual stresses that cause an increase in the surface hardness of the material (CARDOSO; DIAS; ROCHA, 2017; MALISKA, 2003). However, these high stresses can be minimized by preheating the part to be analyzed and potentially reduce the occurrence of cracks (HADAD; EBRAHIMI; ARAEE, 2021).

As far as abrasion cuts are concerned, these can be used on practically all types of materials. The choice of disk and cutting speed parameters depends on the nature of the material to be cut. Thus, materials containing high hardness are cut at high speeds, and discs made of special materials and coatings, such as high-speed steels, tungsten carbide coatings, thin films obtained from nitriding, and diamond-coated discs (AMBADEKAR; CHOUDHARI, 2020; FEDOROV; SHARIPOV; ABROROV, 2021; WANG et al., 2021). In addition, other criteria that influence the quality of the abrasion cut can also be

mentioned: binder hardness; hardness of the material to be cut; velocity; potency; pressure applied to the sample and equipment vibration (ROHDE, 2010). It is also admitted that, because it is a high-quality process and dependent on other factors, it tends to require a longer execution time.

Since material brittleness is directly proportional to hardness, materials with high hardness can also be rapidly sectioned using thermal fracture cutting, such as martensitic steels, for example. One disadvantage of this technique is that thermal cracking may occur on the surface of the sample (CHIAVERINI, 1986a; MALISKA, 2003).In addition to the aforementioned cuts, there are non-traditional cuts, such as laser cutting, water blasting, ultrasonic, electrochemical and electro-discharge cutting. (GLUKHOV et al., 2016; HAN et al., 2021; LLANTO et al., 2021; WETZIG et al., 2019). These have the advantage of high precision, good reproducibility and parameterization, optimum quality, and shorter run time, but require more sophisticated and relatively expensive equipment compared to traditional cutting methods.

In this way, it can be affirmed that the relation "execution time *versus* quality" is inversely proportional, and therefore it should be noted that a proper quality of cut results in a shorter time for performing the sanding and embedding steps on the sample (discussed below), which causes both a boost in the preparation flow and the possibility of making a greater number of samples in a shorter time interval (STEPHENSON; AGAPIOU, 2016).

B. Cleanning

Another extremely important step for sample preparation is surface cleaning, which is essential not to influence the final results of the metallographic analysis (such as: grain size, quantification and phase identification), even preventing residues, oils and coolants from behave adhered to the surface of the sample. With this, possible surface coatings of reaction with the acids of chemical attack, must be removed before the polishing step (ASTM INTERNATIONAL, 2011; COLPAERT, 2008).

For this purpose, alcohol, aqueous solutions, detergents and organic solvents can be used in the manual cleaning stage. Ultrasonic cleaning is recommended in association with hot air jet drying whenever possible. It should be noted that, although it is a more expensive technique (due to the dependence on more modern equipment), it acts with

greater efficiency in cleaning the sample and removing microparticles adhered to the surface of the material (ASTM INTERNATIONAL, 2011; MALISKA, 2003). However, very ductile materials with low hardness (soft) cannot be exposed to long periods in ultrasonic baths as they are more susceptible to cavitation (HOGUE, 2021).

It is also validated the importance of evaluating contaminants through the EDS microchemical analysis instrument together with a scanning electron microscope (SEM) or a Fourier transform infrared analyzer (FTIR), given that on the surface it must be kept only the material to be studied, certifying the use of the characterization methods listed above (ALIYA; ANALYTICAL, 2018).

C. Mounting

Regarding specimens with a small geometry or difficult to handle, the mounting technique is used (coating the specimen with a polymeric matrix) in order to favor the mobility of such samples and improve the performance in the steps of sanding and polishing, as well as preventing the pieces with edges from damaging the sandpaper or the polishing fabric. For the same reason, it is not necessary to embed large parts. (CLAMPS, 2018; ROHDE, 2010).

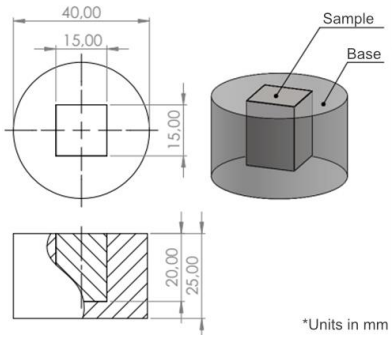
Mostly, two types of mounting are performed, cold and hot. In the first, synthetic resins of fast polymerization are used together with catalysts, admitting a fast polymerization process in a strongly exothermic reaction, and the curing time varies from 12 minutes to 24 hours and depends on the type of material. As far as the second type of mounting (hot mounting) is concerned, it has the advantage of being faster due to the use of thermoplastic materials, and for containing in its polymerization process, a heating and pressure increase by means of presses, commonly known as metallographic mounting machines. (ROHDE, 2010).

In view of this, basically two types of plastics are used, bakelite, a type of synthetic resin formed by polymerization of C_6H_4 e o CH_2O (BAEKELAND, 1909), which, although widely used, is sensitive to long periods of exposure to cutting fluids (GEELS et al., 2007), and lucite, an acrylic material that has characteristics of plastic and glass at the same time, offering a translucent characteristic, allowing a better visualization of the specimen (GEELS et al., 2007; ROHDE, 2010)

In general, a square area with sides of 12 to 15 mm, or diameters of the same dimensions for circular specimens, is recommended for specimen size with mounting.

The height dimensions are preferably limited to 15 to 20 mm. If this is not possible, one should choose a minimum height that is sufficient for correct handling during polishing and grinding, (see Figure 2) (ABNT, 1995; ASTM INTERNATIONAL, 2011).

Figure 2 - Dimensions of specimens for mounting



Source: collection of the authors.

As for the preparation times (heating + cooling), they can be 12 and 15 minutes, respectively for lucite and bakelite, but despite presenting shorter mounting times, they are more easily affected by defects such as circumferential cracks, radial cracks, lack of fusion and "cotton flakes", These defects occur due to failures during the pressing and heating process or part accommodation, and there may even be a compression stress on the specimen due to the non-uniform cooling process of the polymeric matrix, which when excessive, can cause cracks in the material to be analyzed (CLAMPS, 2018).

D. Grinding

The grinding stage occurs to soften the excess material present in the sample, with the intention of flattening its surface and removing the deformations resulting from the sectioning. In sequence, this procedure is usually performed with the help of sandpaper with abrasive grains and hardness higher than that of the material to be worked (VOORT, 2018a). That said, it is presented in Table 1, abrasive grain sizes based on major international standards.

Table 1 - Grit sizes for sandpaper

FEPA*		ANSI#/CAMI+	
Numbering	Size (μm)	Numbering	Size (μm)
P120	125,0	120	116,0
P150	100,0	180	78,0
P220	68,0	220	66,0
P240	58,5	---	---

P280	52,2	240	51,8
P320	46,2	---	---
P360	40,5	280	42,3
P400	35,0	320	34,3
P500	30,2	---	---
P600	25,8	360	27,3
P800	21,8	400	22,1
P1000	18,3	500	18,2
P1200	15,3	600	14,5
P1500	12,6	800	11,5
P2000	10,3	1000	9,5
P2500	8,4	1500	8,0
P4000	5,0	---	---

[#]ANSI: American National Standards Institute
[+]CAMI: Coated Abrasives Manufactures Institute
[*]FEPA: European Federation of Abrasive Producers

Source: (ASTM INTERNATIONAL, 2011)

The grinding can be done manually or mechanized, the latter through rotary motion machines (polishing machines) or linear (APOLLO et al., 2020; SAMUELS, 2003). It is worth mentioning that they are the same ones used in the polishing process, varying only the type of material to be worked (judging the abrasives in suspension on fibrous surfaces of short or long fibrous in the polishing process, as an example, the felt) (GEELS et al., 2007).

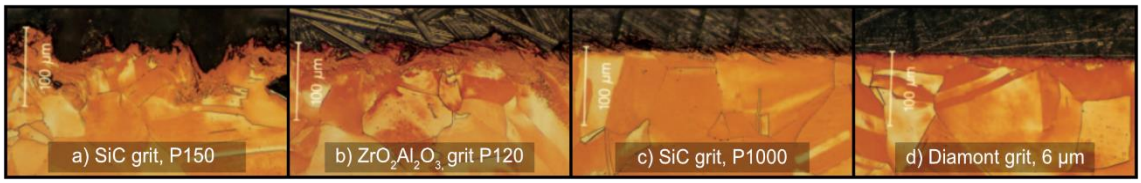
Therefore, depending on the number of machines or compartments available, it is possible to organize the sandpaper in order to obtain the least number of pauses for changing the abrasives and thus reduce the time of the sample preparation process. An example of this arrangement can be seen in the Figure 3.

Figure 3 - Arrangement of sandpapers with different granulometries in the same equipment (manual sander)

Source: collection of the authors

In continuation, the stage is initiated by a "coarse phase", which has as its main objective, the correction of the surface of the specimen as to surface defects and unevenness, for which, sandpapers with a higher grain size are used. Subsequently, fine grinding begins, responsible for reducing the roughness and preparing the sample for polishing (SAMUELS, 2003). The sequence of such grinding can be seen in Figure 4, which in turn shows the surface of a sample after 4 sandings in the above mentioned sequences in different grits (GEELS et al., 2007).

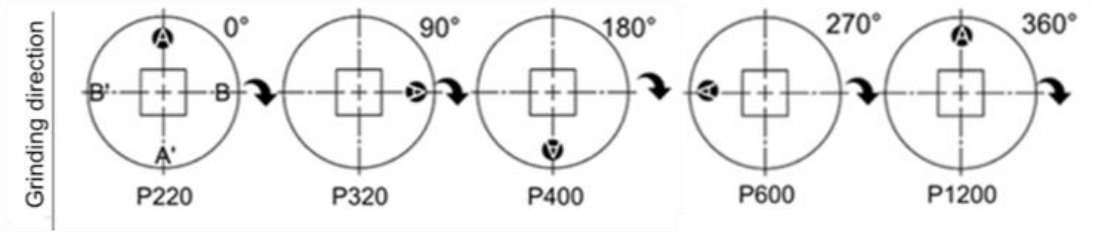
Figure 4 - Side view of the sample in 4 grinding steps



Source: adapted from Geels et. al (2007)

Still by Figure 4, it is observed that as the sandpaper granulometry decreases, the surface becomes increasingly flat. In this sense, there are several recommendations in the literature regarding the granulometry sequence to be adopted, examples being: P180, P220, P320, P400 e P600 (ABNT, 1995) or P120, P220, P320, P400, P600 e 1200 (ROHDE, 2010). However, the gradation depends on factors such as: type of material, hardness, degree of initial deformation, cost, number of samples and time available, judging by those materials of low hardness, whose granulometry ranges can be even larger. In any case, in both processes there must be the presence of lubricant and whenever changing from one sandpaper to another with different granulations, the sample must be rotated in 90° (Figure 5) (ROHDE, 2010; ZIPPERIAN, 2009).

Figure 5 - Representation of the direction of rotation of the sample



Source: adapted from Rohde (2010)

The Table 2 presents the parameters for rotary grinding for a general situation:

Table 2 - Grinding parameters

Step	Size (ANSI)	Lubricant	RPM [C]
1: Planning	P120-400[A]	Water	200-300
2: Fine sanding	P220[B]	Water	200-300
3: Refinement	P500 [B]	Water	200-300
4: Refinement	P1200 [B]	Water	200-300

A: SiC/Al_2O_3
B: SiC
C: complementary rotation, surface and sample rotate in the same direction

Source: (ASTM INTERNATIONAL, 2011)

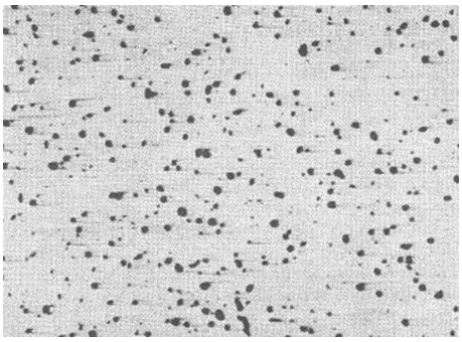
E. Polishing

With regard to the polishing stage, this is different from sanding because it has smaller abrasives (grain sizes equal to or smaller than the order of 6 μm). Its purpose is to obtain a highly reflective surface, free of deformations and scratches (ASTM INTERNATIONAL, 2011) and its execution can occur in the following ways: manual, automatic, vibratory, chemical, and electrolytic.

Mechanical polishing and cleaning of the part during each step must also be done to remove abrasive grains from the previous sanding steps and/or possible contaminants, which prevents the formation of risks that compromise the qualitative metallographic analysis (phase identification) and quantitative (grain size) (COLPAERT, 2008).

In the case of the grinding process, the direction of rotation of the sample influences the final quality of the surface, which must be arranged in the opposite way to the rotation of the plate of the rotating machine (polishing). Thus, the formation of deep unidirectional scratches adjacent to a microstructural discontinuity of the surface of the specimen can be avoided (ASM INTERNATIONAL, 1992; SAMUELS, 2003). This set of risks can also be named in the literature as "comet tails" and is undesirable because it hinders the step of identifying the phases present (Figure 6) (ROHDE, 2010).

Figure 6 - Representation of “comet tails”



Source: adapted from Rohde (2010)

Regarding the quality of the polishing process, this is directly linked to the type of polishing agent (diamond and alumina are the most common) and the type of fabric used in the polishing (short or long hair). Polishing also occurs gradually, always decreasing the granulometry of the abrasive grain, as in the grinding process. (ZIPPERIAN, 2009). The Table 3 presents the polishing parameters for a generic situation.

Table 3 - Polishing parameters

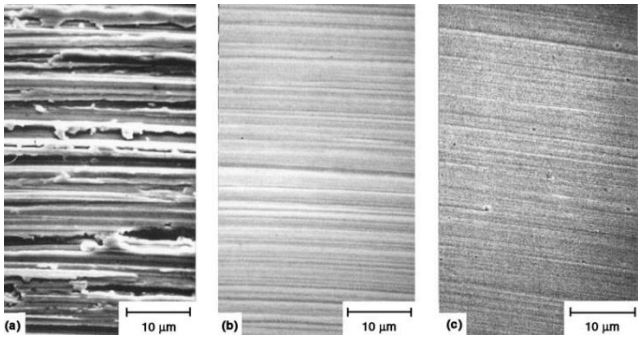
Stage	Size	Lubricant	RPM [CR]
1: coarse	6 μm^*	Water	100-150
2: fine polishing	1 μm^*	Water	100-150
3: refinement	0,05 μm^+	Water	100-150

*: Diamond paste
+: Alumina
CR: counter rotation, surface and sample rotate in opposite directions.

Source: adapted from ASTM (2011) and Rohde (2010)

The result of the effect of the decrease in granulometry can be observed in the Figure 7.

Figure 7 - Effect of varying the granulometry of the polishing agent. (a): sanded surface (b): first polish with 6 μm diamond paste; (c): second polishing with 1 μm diamond paste



Source: (SAMUELS, 2003)

It can be seen from the Figure 7 that the surface roughness reduces as the granulometry of the abrasive for polishing decreases, in this way the surface tends to become smoother.

F. Etching

Further, the microstructure of various materials is only revealed after the chemical etching of the surface (contrast) of the sample, which is performed by immersion or

smearing, admitting most often for carbon steel alloys, the reagents Nital (nitric acid plus alcohol) and Picral with an immersion time ranging from 2 to 15 seconds. In addition to these reagents, other solutions are also included, each with a specific purpose and material (Table 4) (LENDA et al., 2020; SAMUELS, 2003; SCOTT, 1991).

Table 4 - Other common reagents for chemical etching

Reagent	Composition	Application
Oberhoffers	500 ml of distilled water, H_20; 500 ml of ethanol, C_2H_5OH; 42 ml of HCl; 30 of $FeCl_3$ 0,5 g of $SnCl_2$	Segregation studies
Heyn	20 ml of distilled water; 20 g of CuCl (NH_4)	Phosphorus segregation
Klemm	50 ml of $Na_2S_2O_3$; 1 g of $K_2S_2O_5$	Phosphorus segregation
Baumann	100 ml of H_2O; 5 ml of H_2SO_4	Arrangement, distribution and inclusion of Fe and Mn.
Beraha	100 ml of H_2O; 24 g of $Na_2S_2O_3$; 3 g of citric acid; 2 g of cadmium chloride	Ferrite contrast
Alkaline picrate	2 g of picrate acid, 25 g of NaOH; 100 ml of H_2O	Distinguish between carbide and ferrite
Marshall	5 ml de H2SO4, 8 g of oxalic acid and 100 ml of water	Austenite contrast
Beaujard	20 g of sodium bisulfide; 100 ml of H_2O	Hardened ferrite and martensite
Vilella	2 g of picric acid; 100 ml of ethanol; 5 ml of HCl	Austenite contrast
Whiteley	5 g of $AgNO_3$; 100 ml of water.	Sulfide Inclusions

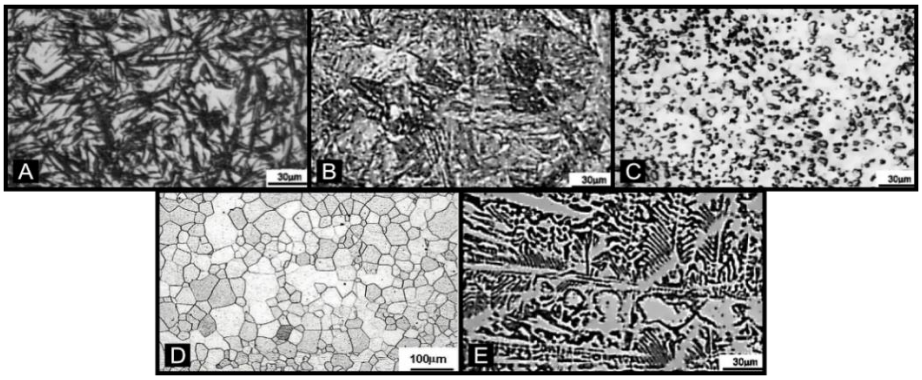
Source: adapted from several authors (AGUIAR et al., 2008; LENDA et al., 2020; SCOTT, 1991; VOORT, 2018b)

Finally, immediately after the etching, the surface should be washed with alcohol and cotton, and then dried with hot air. As these are mostly acid reagents, all handling must be carried out in a controlled environment and with adequate personal protective equipment to prevent accidents to the operator (EQUIPMENT; WEIGHTS, 2010).

EXPECTED RESULTS IN THE ANALYSIS

microstructure of the material, but also its present phases and the existence of defects, all of which are important information to characterize the material and ensure its processing quality. (SCOTT, 1991). As already briefly mentioned, it can be performed on devices (optical microscopes) that use a certain number of lenses for reflection magnification of the contrasted image of the sample surface. The parameters of illumination, lens, sample positioning are properly adjusted for each magnification level, surface type and magnification level (ROHDE, 2010). It can be seen, therefore, Figure 8, some examples of microstructures revealed from the microscopy examination.

Figure 8 - Examples of Microstructures



Source: (ABNT, 2007)

In short, the Figure 8.a denotes a representation of the retained austenite phase (light color), in turn being a component to be controlled, since it deteriorates the mechanical characteristics of tempered steel, for example, negatively implying in its dimensional properties, reduction hardness and mechanical strength. Finally, the predominant martensite phase (dark color) is also visible, which, on the other hand, increases the mechanical performance of case-hardened steels. (FONSECA et al., 2021).

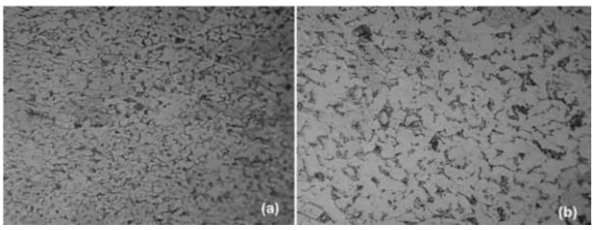
As the Figure 8.b suggests, the bainite phase can be evidenced, a transformation product formed in an intermediate temperature range between the eutectoid and martensite transformation, consisting of ferrite and cementite aggregates (COLPAERT, 2008). In Figure 8.c, the spheroidized Cementite phase is observed, identified by “small craters” on its surface. (COLPAERT, 2008). It is worth mentioning that the purpose of the heat treatment called spheroidization, which proposes this phase as a result, is to generate a microstructure of globular carbides in a ferritic matrix, when the priorities are low hardness, high ductility and better machinability (ASM INTERNATIONAL, 1964).

Regarding Figure 8.d, the ferrite phase is observed, arranged in eutectoid steels with irregular grain structure and with properties of low hardness, low tensile strength, satisfactory shock resistance and high elongation, also bearing ductility (CHIAVERINI, 1986b). Finally, in addition to the aforementioned phases, the Figure 8.e admits the phase of Ledeburite, which is a eutectic mixture typical of a cast iron (FISCHER, 2008).

Therefore, in the figures presented above, very precise enlargements are demonstrated, each microstructure has particular characteristics observed in the micrograph. These images would not be possible without the aid of optical microscopes and or if they were poorly prepared surfaces.

Further, in a study involving SAE 1020 and SAE 1060 steels, after the metallographic cutting procedures, hot mounting, identification with the aid of a pyrograph, grinding in a polishing machine using silicon carbide sandpaper (admitting seven different granulometries), polishing also in a polishing machine with granulations of 1µm, 0.3µm to 0.5 µm and etching by 3% Nital with an interval of 3 seconds (steps covered in this work), it was obtained from an optical microscope under magnifications of 20 and 40X, the constituent phases of ferrite and pearlite (Figure 9), in addition to proving the amount of carbon corresponding to the nomenclature of the samples in question (RODRIGUES et al., 2014).

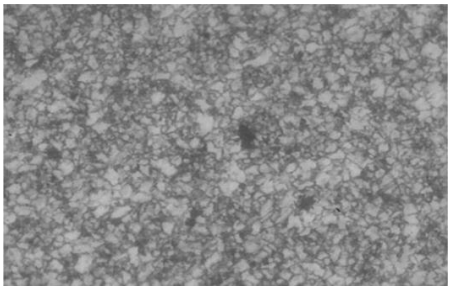
Figure 9 - Metallographic analysis after SAE 1020 steel sample preparation procedures under magnifications of (a) 20X and (b) 40X



Source: (RODRIGUES et al., 2014)

Similarly, in a research demonstrating the microstructure of the same steel (SAE 1020) and after the same sample preparation procedures, the sectioning dimensions resulted in 9.525 mm x 12 mm, being subjected to heat treatments in muffle furnaces followed by cooling for the purpose of change its mechanical properties (not discussed in this work), however, the same phases previously mentioned were also identified under the sample without heat treatment, this time with an increase of 400X magnification (Figure 10) (FREITAS et al., 2016). It is worth noting that the Ferrite microconstituent phase on the grain boundary appears clearly, whereas the Perlite phase appears darkly (RODRIGUES et al., 2014).

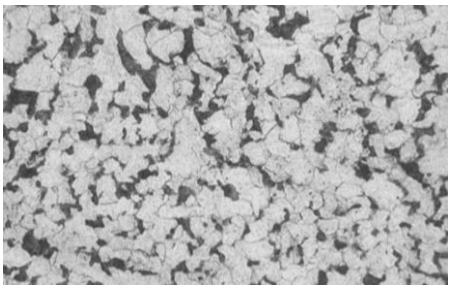
Figure 10 - Micrograph of 1020 steel under 400x magnification



Source: (FREITAS et al., 2016)

Similarly, in another study performing metallographic examination on the same material (SAE 1020 - see Figure 11) and with the same cutting procedure, mounting, grinding under water lubrication against the grain size in the sequence of: 600, 800, 1000 and 1200 mesh and polishing in polishing machine with alumina and diamond paste abrasives followed by Nital attack at 3% and drying with hot air jet, it was proven in its microconstituents the cementite phase in 3% (shown by the dark section) and ferrite in 97% (shown by the green portions), results from a quantitative analysis associated with qualitative (LEITE et al., 2017).

Figure 11 - Micrograph of SAE 1020 steel



Source: (LEITE et al., 2017)

It is validated, therefore, a pattern of micrographs in relation to the same material (SAE 1020), although in the three aforementioned researches these have been given in different qualities, this is a result of the different sample preparations, ratifying again the importance of these procedures regarding the precision and identification of its microconstituents.

CONCLUSIONS

It was found from the literature review that the sample preparation phase is extremely important for the quality of metallography analyses, and must be carried out with care and safety, both in terms of handling, available time, quality and cost.

It was also verified that, in the sanding and polishing stages, there are well-defined processes, although depending on the material, there may not be a protocol, which is adjusted from a new or existing one, or from works already carried out with the same stuff. In addition, the main techniques for preparing metallic samples were presented, so that a

preparation sequence (flow) was developed that can be used in most carbon steel alloys and general applications.

Finally, it is noteworthy that the sample preparation steps must be carefully performed, in order to eliminate possible flaws and defects that could compromise the microscopy analysis.

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Recebido em: 12/03/2022

Aprovado em: 23/04/2022

Publicado em: 28/04/2022