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Microscopy Techniques to Analyse the Characteristics of the Metallic Materials

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Microscopy techniques are powerful tool to evaluate the characteristics of the metallic alloys, as microstructure, chemical composition and crystalline orientation. Optical Microscopy (OM) is technique that allows analysing the microstructure and crystalline orientation of these materials. This microscopy technique is characterised by fast processing, low cost and being non-destructive. The imagines of OM are obtained via the light reflexion or transmitting with the material. Although optical microscopy is an excellent analysis technique, it presents certain limitations, as the inability to determine the chemical composition of material. Scanning Electron Microscopy (SEM) can rapidly analyse the microstructure and the chemical composition of the metallic alloys with a high resolution. SEM uses accelerated electrons to get pictures of the metallic materials features. Accelerate electrons interact with the atoms and electrons from surface layers of the material, which generates diverse types of the electrons and radiations. According to the type of electrons or radiation, different kinds of the materials characteristics can be seen. This technique provides good imagens of the characteristics of the material without producing damage, though the crystalline orientation is non-determinable via SEM. For this reason, both analysis techniques are complementary. Although OM and SEM are important tools to evaluate the metallic alloys, these materials must be pre-treated to be able to be characterised. The pre-treatment comprises of a mirror-to-polish and subsequent, etching process that defines the type of visible characteristics of metallic materials. In this chapter, the pre-treatment, OM and SEM to analyse the characteristics of the metallic alloys will be discussed.

Keywords: *Optical Microscopy; Scanning Electron Microscopy; Mirror Polish; Etching Process and Metallic Alloys*

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1. Introduction

Optical and electron microscopy are great techniques to evaluate the characteristics at micro and nano-scale of metallic materials, as topography, microstructure and chemical composition. These methods stand out from others because their excellent properties as, non-contact, friendly with the environment, non-destructive, fast and viable at different conditions (Vander Voort 1999). Optical and electron microscopy are complementary techniques to each other. OM can determine the grain orientation metallic

materials (SCOTT 1991) while SEM can define the topography (secondary electrons) (Vernon-Parry 2000) and chemical composition (backscattered electrons) (Zhou et al. 2006) of metallic alloys. SEM has a resolution at nano-scale while OM resolution only is at micro-scale. Images of the OM can be obtained in colour while SEM pictures are in grey scale. Besides, both techniques can also evaluate the topography and microstructure of alloys at micro-scale. However, the metallic materials must be previously treated to analyse their microstructures. Pre-treatment is consisted of two parts; polishing and etching. Polishing process has to be carried out before etching. The objective of the polishing

is to remove any surface imperfection (e.g. scratch, pitting or corrosion) that can hinder or block the correct analysis of the microstructures (Vander Voort 1999; SCOTT 1991). Etching process should be conducted within few minutes after polishing process to have the best possible results. Different metallic materials features can be observed according to the etching conditions, as such gran orientation, second phase structures, carbides and etc.(SCOTT 1991; Vander Voort 1999; Bramfitt and Benscoter 2002; Petzow 1999).

In the present chapter, the features of the pre-treatments, OM and SEM analyses on metallic alloys are described. The pre-treatments for metallic materials are commented to beginning of the chapter. OM characteristics for analysing the alloys are studied in the next section. In the last section, SEM features about evaluation of the metallic alloys are described.

2. Pre-Treatments

Previous treatment to microscopy analysis is formed by two steps: polishing and etching. The polishing treatment should be carried out before etching process. For this reason, polishing process is described before etching treatment in this section.

2.1. Polishing

Polishing is defined as the process to generate a smooth and shiny surface via mechanical, chemical, electrolytic or electrochemical methods. Polished surfaces usually have a high specular reflection, which is limited by the refraction index of the material (Faust 1943; Swihart 1953; Landolt, Chauvy, and Zinger 2003; Der 1950; Samuels 2003). The peak and valleys of the material surface are removed through the polishing treatment and surface roughness is therefore reduced. Although surfaces can be considered as polished when its average roughness are $\leq 600\text{nm}$ (EN), metallic materials should be polished at average roughness $\leq 40\text{nm}$ to have the best result in etching process. Polishing can be carried out via chemical, electrical or electrochemical method, but mechanical method is recommendable for polishing the metallic samples because this method generates surfaces with lowest average roughness and imperfections.

Polishing is produced via rubbing the surface with harder materials (SCOTT 1991; Samuels and Consultants 1992; Leng 2013). This method is composed of two steps; grit and cloth. First, grit step must firstly be conducted and then, the cloth step has to be done. Grit step can be carried out via two different methods, abrasive blasting and grit paper. A stream of the abrasive materials forcibly ejected on rough surface is used in the abrasive blasting (Achtsnick et al.

2005; Mills 2014). This method can be typified according to the kind of the abrasive element. The average roughness of the surface is defined by the type of the method, as can be seen in Table 1.

Table 1. Diode parameters obtained via thermionic emission theory.

Name	Abrasive Material	Average Roughness (μm)
Sandblasting	Sand	≥ 2.500 (SLĂTINEANU et al. 2011)
Wet abrasive blasting	Vapour	≥ 4.000 (Careddu and Akkoyun 2016)
Bead blasting	Fine glass	≥ 0.800 (Vecom)
Shot blasting	Sand	≥ 2.700 (Rodríguez-Hernández et al. 2011)
Hydro-blasting	Water	≥ 12.000 (Draganovská et al. 2018)
Micro-abrasive blasting	Glass (10-150 μm size)	≥ 0.100 (Melentiev et al. 2019)
Dry-ice blasting	Carbon dioxide (solid)	≥ 1.400 (Uhlmann 2008)
Bristle blasting	High carbon-steel	≥ 16.000 (CUDIC 2019)

Grit paper method is carried out via abrasives paper that can be silicon carbide, zirconium or diamond, but abrasive papers commonly used to polish metals are silicon carbide. Abrasive papers are classified through the grade that indicates silicon carbide grain/inch (Sin, Saka, and Suh 1979; Orvis and Grissino-Mayer 2002; Zipperian 2001). Surface average roughness is determined by the grade of the paper. Lower roughness can be obtained with paper of high grades. Table 2 shows examples of average roughness can be achieved according to abrasive paper grade.

Polishing via grit paper method should be begun at abrasive paper of low grades and subsequence increasing the grade of the abrasive papers at every step. This method can carry out manual or automatic way. In the case of the manual way, the direction of the polishing must be perpendicular to the direction of the previous polishing step (Dmitri 2003), can be illustrated in Figure 1.

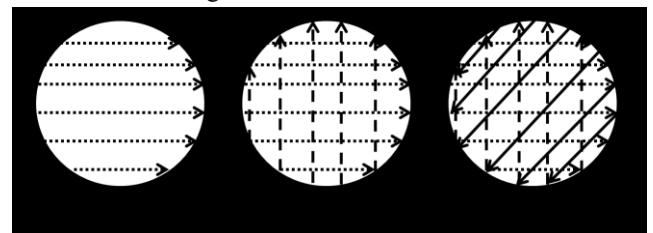


Figure 1: Schematic drawing of the manual polishing method via grit paper method.

Table 2: Average roughness according to abrasive paper grade (Zipperian 2002; Orvis and Grissino-Mayer 2002).

Grade of paper (European P-Grade)	Average roughness (nm)
P80	1140
P120	1050
P180	880
P220	300
P360	230
P800	120
P1200	110
P2400	25
P4000	20

On the other hand, the automatic method should be conducted with polishing machine and under lubrication of water to avoid the corrosion of the samples (Samuels 2003; Evans et al. 2003).

Although abrasive blasting is a fast process that removes the most of the imperfections, finished surface has unreasonable average roughness for going to the cloth step. For this reason, post-grit with high grade abrasive paper ($\geq P1200$) of the blasted surface is recommendable to reach adequate average roughness, which usually is ≤ 110 nm.

Cloth step is generally conducted with a soft cloth and a chemical product that usually is diamond paste or dissolution of the colloidal silica gel. Cloth step must be also carried out by mean of mechanical polishing machine and under lubrication, independently of the chemical product. Diamond paste comprises of mono or polycrystalline diamond dissolved in oil base (Samuels 2003). Monocrystalline diamond pastes usually produce surface finish with lower average roughness than polished surface through polycrystalline diamond pastes (Samuels 2003). The lubricant of the diamond paste commonly is a dissolution of methylene blue in alcohol (isopropanol or ethanol) (Samuels 2003). Crystal size determines average roughness of the polished surface, can be observed in Table 3. Average roughness obtained via paste diamond polishing is lower at smaller crystal size (Samuels 2003). Surface finish of diamond past at $3\mu\text{m}$ satisfies the average roughness conditions to carry out the etching. However, it is recommendable to conduct a last polishing via colloidal silica gel dissolution to obtain the best results in optical and electron microscopy.

Polishing with colloidal silica gel dissolution can eliminate the imperfections at micro and nano-scale on metallic surface, as pitting and scratch. Cloths for this polishing usually are suitable for chemical products (Samuels 2003). Colloidal silica gel dissolution comprises of colloidal silica gel and polar liquid (dissolvent). Colloidal silica gel can be of 0.060 and $0.040\mu\text{m}$ of size grain (Sivanandini, Dhama,

and Pabla 2013). Polar liquid is commonly distilled water, an exception some metallic materials as titanium or gold. In these cases, distilled water is replaced by hydrogen peroxide. This type of the polishing is considered as chemical-mechanical polishing (Evans et al. 2003; Sivanandini, Dhama, and Pabla 2013). In both case, dissolution commonly is 1:1 colloidal silica gel: polar liquid. Polished surface via this method can present an average roughness around $0.005\mu\text{m}$. For these reasons, it is recommendable to terminate the polishing process of the metallic samples with colloidal silica gel dissolution. An example of the 316L stainless steel polished surfaces via diverse abrasive papers, diamond pastes and colloidal silica gel can be viewed in Figure 2.

Table 3: Average roughness according to crystal size of the diamond paste (Sinha 2006; Samuels 2003).

Crystal size of diamond paste (μm)	Average roughness (μm)
15	0.100
6	0.050
3	0.025

2.2. Etching

Although some microstructures of some metallic materials can be seen only with mirror polishing, all microstructures of all metallic alloys can be observable via etching process (Petzow 1999; Leng 2013; Louthan Jr 1986). This process highlights the features of the alloys at microscale (Vander Voort 2007). This is possible because the control of the corrosion process, which is generated during etching, via the different corrosion potential of the element from metals (Vander 2012; Vander Voort 2007; Dmitri 2003). Etching can be carried out via different methods that are classified as, chemical, electrochemical, thermal, plasma, molten salt and magnetic (Zipperian 2001; Vander 2012).

Chemical etching is a most common used etching, which uses aggressive dissolutions to conduct the development of the microstructure. Dissolution commonly is strong acids or bases and reducing or oxidising element though alcohols can be used in the etching (Vander 2012).

Electrochemical etching is another method most used and combines aggressive dissolution with potential or current. A cathode, an anode and electrolytic bath form the elements of the electrochemical etching. Anode and cathode should be same metallic material and the anode is the etched sample (Węglowski 2013; Petzow 1999; Zipperian 2003).

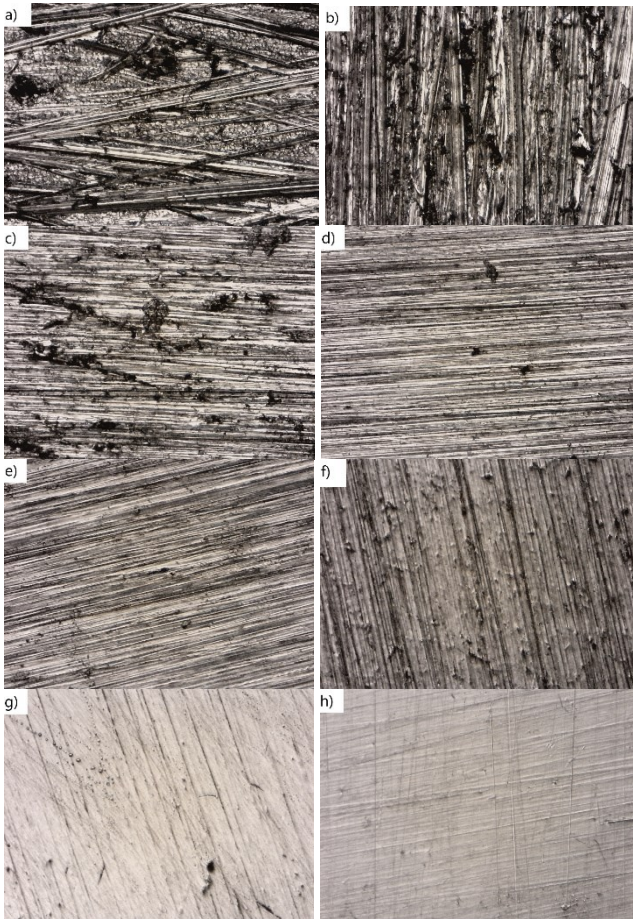


Figure 2: Figure 2. OM pictures of 316L stainless steel polished with a) P80, b) P120, c) P160, d) P320, e) P600, f) P1200 SiC paper, diamond paste of g) 6µm, h) 3µm and i) 1µm size and, j) 0.04µm colloidal silica gel.

Electrochemical etching is another method most used and combines aggressive dissolution with potential or current. A cathode, an anode and electrolytic bath form the elements of the electrochemical etching. Anode and cathode should be same metallic material and the anode is the etched sample (Węglowski 2013; Petzow 1999; Zipperian 2003).

Thermal process etches the samples via high temperatures and at low pressure. Samples etched through thermal etching usually have coloured grains, second phase and intermetallic. Although thermal etching is commonly used in ceramography, some metallic alloys as austenitic stainless steel, can be etched via this method (de Andrés et al. 2002; Chinn 2002; Petzow 1999).

Plasma etching involves a high speed stream of light discharges that is shot on samples. Etchants comprise of adequate mixed gases that commonly are charged (ion) or neutral (atoms or radicals) (Hull, Leonhardt, and Sanders 1992). This method is rarely used in etching of the alloys because it is complicated and expensive.

Molten salt etching is conducted at high temperature and with electrolytic chemical compounds (salts). Temperatures used in this process should be sufficient to melt the salts

(Zipperian 2001). Although this method is utilised to develop ceramic and glass, some metallic materials can be etched through molten salt etching.

Magnetic etching applies a magnetic field about colloidal magnetic particles that are etchant (Gray 1974). This process provides high reproducibility and precision though it is expensive and complicated.

Chemical and electrochemical methods are most common etching used for metallic materials due to their good characteristics, as simple, fast and cheap. According to the etching conditions, one or other microstructural features of the metallic alloys can be developed. It is important to note that the grain orientation can be only visible via polarised light (E407-07 2015). Table 4 lists examples of the developed microstructural characteristics depending on the alloy and the etching parameters.

Chemical and electrochemical etchings are conducted with aggressive dissolutions that must be neutralised before the surface analyses of the microstructures. Strong acids or oxidisers should be neutralised via bicarbonate whilst acetic acid is recommendable to neutralise the strong bases or reducers. In addition, etched samples should be cleaned before the optical and electron microscopy analyses. Also, it is commendable to carry out the surface analyses at few minutes after etching. If it is impossible, etched samples should be kept in desiccator to avoid the corrosion of the surfaces. In the case of the corrosion or burnt of the samples, surfaces should be polished and etched again.

3. Optical Microscopy

It is surface analysis method via optical microscope that uses visible light and system of lens to magnify of microelements (Murphy 2001). The first optical microscope was created by Zacharias Jansen in Middleburg, Holland at 1595 (Masters 2001). Optical microscopy is utilised in several areas as, medicine, biology, metallography and chemistry, because its excellent properties, such as low cost, easy to use, viable for life cell and in-situ (Davidson and Abramowitz 2002; Spring and Inoué 1997; Wolf and Sluder 1998). Although optical microscopy is a technique that can carry out pictures at high magnification, it is limited by its poor resolution at elevated magnifications. Airy disk can happen at very high magnifications with transmitted light. This event is characterised by diffused circles with diffraction rings, which reduce the resolution of the optical microscopy. The resolution, d , is therefore determined by wavelength of the light, λ , and Number Apertures, NA , as can be viewed in 1 equation (Schmolze et al. 2011; Jonkman et al. 2003).

$$d = \frac{\lambda}{2 \cdot NA} \quad (1)$$

In last decades, several researchers have worked to break the resolution limit. This continues to be an important limitation

of the optical microscopy though great advances have been reached in the recent times (Schmolze et al. 2011).

In the field of the surface analysis of the metallic materials (Metallography), optical microscopy is a powerful and traditional tool that allow to evaluate the structures, phase, intermetallics and grain orientation of the metallic alloys at micro-scale (Vander Voort 2007; Zipperian 2011). However, the parameters of the optical microscope and etching dissolution (Etching subsection) define surface features of metals that can be visible. For this reason, the following subsections detail the characteristics of the optical microscope, types of optical microscopy and applications on the metallography.

3.1. Optical Microscope

Traditionally, optical microscope is called as optical light microscope because it utilises light to conduct the analyses of the elements. Optical light microscope comprises several items that allow doing the surface evaluation of the samples. These items are eyepiece, revolver, objective lenses, diaphragm, condenser, light source, coarse and fine adjustments. Figure 3 illustrates the items of the optical light microscope.

Eyepiece is utilised to bring the focused image for eyes and different objectives can be used with the same eyepieces (Abramowitz 1985). Revolver allows selecting the diverse objectives lenses of the optical microscope, e.g. 5X, 10X, 20X and 50X (Davidson and Abramowitz 2002). Objective lenses are items that collect the light from the sample surface. These items are cylinder that contain single or multi compounds glasses as lenses. Currently, objective lenses are focal lens, which allow staying focus when the focal distance or magnifications are changed. Characteristics of the objective lenses are magnification and NA. Special types of the objective lenses are oil-immersed and water-immersed objectives, which are used when the materials have higher reflectivity index than air (Malureanu, Ferrari, and Di Fabrizio 2004). The condenser is a lens to focus the light onto the samples. Diaphragm can be added to condenser and its function is to improve the quality and intensity of the illumination (Vainrub, Pustovyy, and Vodyanoy 2006). The light source can be wide range of types, as bulb, LEDs, laser, halogen lamps, etc....(Wegerhoff, Weidlich, and Kassens 2007). Course and fine adjustments are items to move the objective lenses and for focus the images.

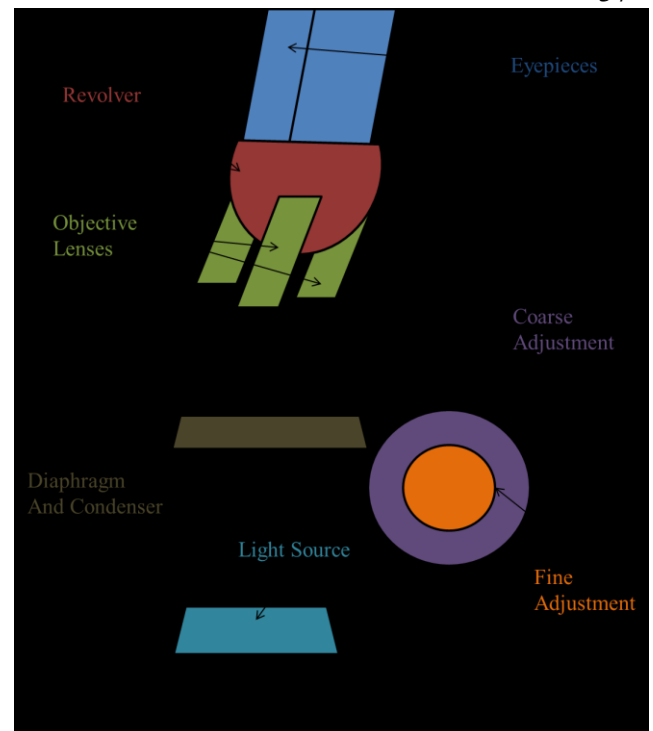


Figure 3: Schematic drawing of the optical microscope.

Other items can be included in the optical light microscope, such as capture images device and light modifier device. Capture images device is a system that allows obtaining images from the optical light microscope. The first capture images devices were photographic films but digital camera. Complementary Metal-Oxide-Semiconductor (CMOS) and Charge Couple Device (CCD) currently are the most used devices. Even, CCD has permitted to produce the truth digital microscope that is free of eyepiece (Davidson and Abramowitz 2002; Centen 2014). These devices can also obtain videos from optical light microscope, which are very useful for biological and medical field. Light modifier device is a system that permits to change the light features. This device can be polariser, paraboloid, cardioid, phase shift and grey filter rings. These items are related to the types of optical microscopy and consequently they will be argued in the next subsection.

3.2. Types of the Optical Microscopy

Types of the optical microscopy are classified according to the characteristics of the light, as discussed earlier in this chapter. One optical light microscopy type is more adequate than others depending on the material features or the characteristics that are wanted to evaluate. Main types of the optical light microscopy are multi-colour, bright field, dark field, cross-polarised light and phase contrast (Davidson and Abramowitz 2002; Vander Voort 2007). Figure 4 shows OM images of tissue paper obtained via different OM methods.

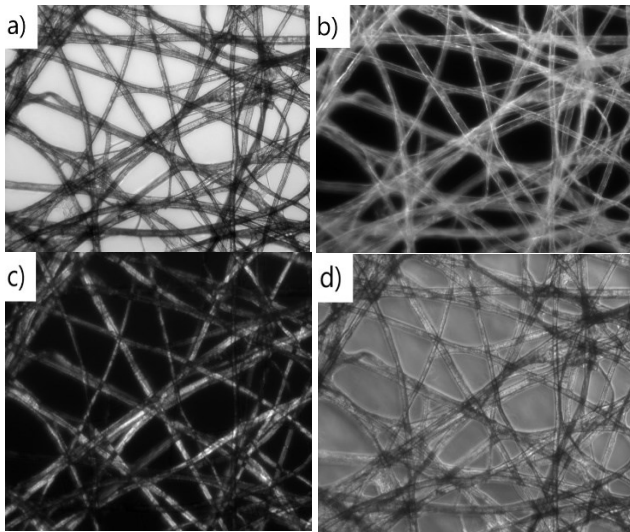


Figure 4: OM images of the tissue paper at 10X magnifications through bright-field a), dark-field b), cross-polarised light c) and phase contrast d) (Zephyris 2010).

Multi-colour utilises visible range light (from 400nm to 700nm) and allows distinguishing the different colours of the microstructures. Extra items are non-necessaries for this method. Etched samples with microstructures of different colours are commonly analyses via multi-colour optical light microscopy (Wegerhoff, Weidlich, and Kassens 2007). This type is inadequate for most of the etched metallic samples or for samples with similar colours microstructures. Bright field employs white light (from 560nm to 600nm), which improves the contrast of the materials characteristics (Wegerhoff, Weidlich, and Kassens 2007). High contrast is achieved by the attenuation of the transmitted light in dense areas of the samples (Leng 2013; Bagnell 2012; Dmitri 2003). Images obtained via bright field are in grey scale. Transillumination light source is the necessary extra-item to carry out this type of optical light microscopy. Halogen lamp usually is utilised as transillumination light sources (Bordo and Rubahn 2005). Iris diaphragm, oil-immersion objective and polarising filter can be added to optical microscope to improve the quality of the pictures (Bagnell 2012). Although this method is widely used to analyse etched metallic alloys, it present certain limitations as, 1300X maximum magnification, low optical resolution and inviable for transparent or colourless materials. For these reasons, bright field is unpopular method to analyse the biological materials.

Dark field uses the same light than the bright field but, in this case, the full cone of light is replaced by hollow light cone. This produces that the light from the sample travels around objective lenses, and the samples therefore are invisible in the pictures of the dark field (Bagnell 2012; Villiger, Pache, and Lasser 2010). Filter holder is used to carry out this type of method and it is localised between light source and sample. This element is formed of direct illumination block to scatter light and patch stop to block

the light (Laronga and Thorburn 1993; Dmitri 2003). This is commonly applied to characterise the transparent materials, as biological cells (Bagnell 2012). This method is only utilised in metallography field to evaluate fractures in alloys. Crossed-polarised light permits to analyses optical features of the birefringence materials through light polarised white (rlton 2011; Swann and Mitchison 1950). Images form this method usually exhibit different contrast according to interaction of the sample with the polarised light. Material modifies or removes certain wavelength due to its electromagnetic field (Chayen 1983; McCrone 1994). In the specific case of the metallic materials, the plasmon is the responsible of this effect. In addition, the thickness of the elements from samples can be defined via this method. The modification of polarised light when crosses a birefringence material is called as Optical Path Difference (O.P.D.). O.P.D is defined by λ and refraction index of the material's elements (n_e), as can be observe in 2 equation (Frandsen 2016).

$$O.P.D = 2 * \pi * \left(\frac{(n_e - n_o) * t}{\lambda} \right) (2)$$

Being, n_o the refraction index of air and t the thickness of the elements. Nonetheless, n_e must be known to determine t and vice versa.

Extra devices utilised to conduct this method are a polariser glass and analyser. Polariser glass is localised after the light source whilst analyser is placed before eyepiece. The analyser also is a polariser glass that improves the resolution of the optical microscope (Frandsen 2016). Additionally, this method can be combined with dark field by inclusion of the direct illumination block (Vander Voort 2007). The combination of this method with adequate etching dissolution (Etching section) permits to analyse the microstructural orientation of the grain, second phase and inter-metallics from the metallic alloys (SCOTT 1991). Moreover, the polarised light method can be utilised in mineralogical and biomedical field for evaluating minerals, bones, teeth or urine crystals (McCrone 1994). This method is non-useful for materials with same crystalline orientation like cell or metals without grain (SCOTT 1991).

Phase contrast allows observing the microstructures with dissimilar refractive index. The diverse wavelengths of the light suffer different delays to pierce the sample. These delays produce a waves being 'out of phase' with others, which can be transformed into amplitude differences that is seen as brightness variation (Liang, Erwin, and Mansuripur 2000). Extra devices of this method are -90° shift ring and grey filter ring, which are placed after objective lenses. -90° shift ring is utilised to scatter the light from sample while grey filter ring is used to improve the contrast (Maurer et al. 2008). For this reason, images of this method commonly are in grey scale. This type of optical light microscopy is applied to characterise transparent samples. In the past, it was utilised to evaluate transparent minerals but nowadays, it has

been replaced by other techniques as, Transmission Electron Microscopy (Heaysman, Pegrum, and Preston 1982). However, this method is unable to be utilised to examine living specimens, e.g. cells and micro-animals, due to these living organism die at low pressure that generated the vacuum system (Mann et al. 2005).

Other variants of the optical microscopy exist but they need an intrinsic modification of the optical microscope, as light position, detector, non-visible radiation, lenses and etc.... Therefore, a detailed analysis of these methods is not described in this chapter. However, short summary of these techniques can be viewed in Table 5.

3.3. Metallurgical Application of the Optical Microscopy

Optical light microscopy is widely used in metallographic field to determine the microstructural characteristics of the metallic materials, such as grains, second phases, and inter-metallics. Besides, this technique allows also evaluating other surface features, as micro-cracks, pitting, fractures and etc.... (Louthan Jr 1986). SEM is a great tool for the metallography as well, but OM remains as main technique for defining the microstructures of the metallic alloys. This is due to a certain limitations that are presented the electronic microscopy. Nevertheless, OM and SEM are complementary techniques in the area of the metallography (Borel et al. 2014).

Objective lenses utilised in the metallography commonly are from 50X to 2000X though other magnifications can be used to this aim (Vander Voort 2007). The metallographic analyses use reflected light and illumination light should be therefore placed on vertical position. In addition, the light should perpendicular arrive to the surface sample for avoiding shadows or other effects that could influence in the optical microscopy analysis (Dmitri 2003). Main OM techniques are multi-colour, bright field and polarised light. Other types of the optical light microscopy could be utilised in the metallurgy but only in specific cases, e.g. low contrast or optically anisotropic materials (Vander Voort 2007).

Multi-colour is commonly utilised on etching samples that have natural colour microstructures. Etching dissolution can be different types (thermal, chemical or electrochemical) because the natural colour depends on features of the metallic alloy. However, thermal etching usually colours the samples (Vander Voort 2004; Zakerinia, Kermanpur, and Najafizadeh 2009). Pictures can be obtained colour reversal film or negative colour reversal film. Colour reversal film is cheap whilst negative colour film has better quality and can be in white and black (Vander Voort 2007). Grains of the matrix are seen as polygonal structures of varies colours while homogeneous matrix shows the same colour in all structure. Second phases are distinct colour than the matrix or grains and their shapes usually are polyedrical. Inter-

metallics also have dissimilar colour than the material matrix but their morphologies commonly are spherical or oval (Vander Voort 2004). Examples of the images carried out via this method can be observed in Figure 5. Although this method is able to be applied on other etched surfaces, this is non-recommendable because the pictures can lose contrast and definition.

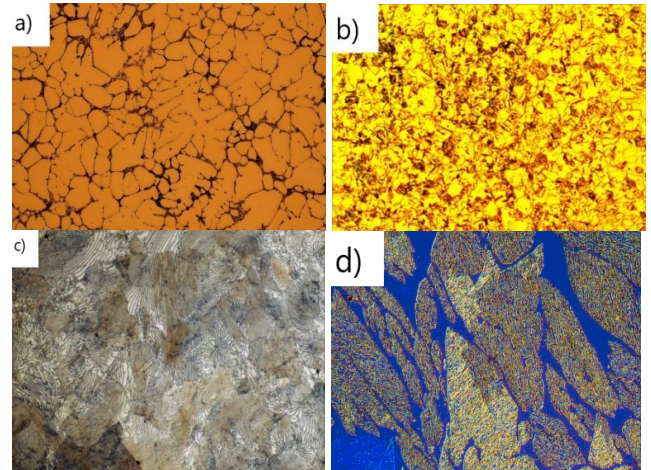
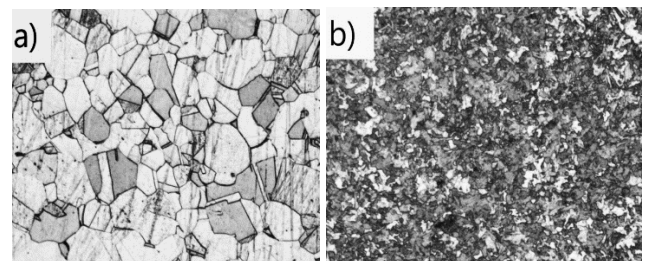


Figure 5: Multicolour OM images of a) copper, b) Hallestolly, c) 1095 high carbon steel and d) 431 stainless steel (Technologies 2019) Copyright 2006-2019 PACE Technologies ®.

Bright field is the method most widely utilised to analyse the microstructures of the metallic alloys. This is due to the excellent quality and high contrast of the metallographic pictures (Vander Voort 2007). The different microstructures from metallic samples are seen with different tones in the grey scale range. Homogenous matrixes are white without line while grains are differentiated by the grain borders. Grains are white whilst the borders are black because these are zone easier to be attacked by etching process. Second phases and inter-metallics usually have different hues than matrix of the metallic materials, but second phases commonly are polygonal structures while inter-metallics have circular shapes (Vander 2012).



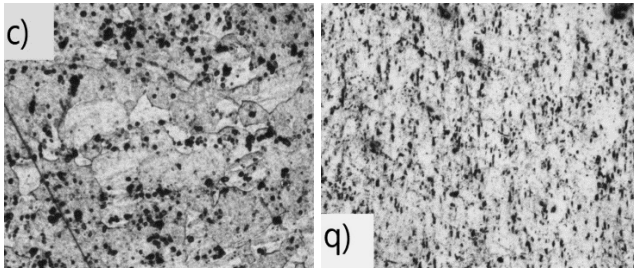


Figure 6: Bright-field OM pictures of the general microstructures of a) 316L stainless steel, b) 355 steel, c) 6061 and d) 3103 aluminium alloys at 1.5X magnifications. Stainless steel and steel were etched by 88 etching dissolution according to ASTM E407-99 (E407-07 2015) Aluminium alloys were etched by 3 etching dissolution according to ASTM E407-99 (E407-07 2015).

Polarised light is applied to evaluate grain orientation and to analyse the microstructures of the metallic materials that are difficult to etch (Vander Voort 2004; Vander 2012; Calvo, Gautier, and Simon 1991). In some case, the samples should be etched with specific dissolution (Etching. section) for making visible the grain orientation of the metallic alloys. Polarised light from isotropic metals (amorphous crystalline structures) are non-contrast because reflected or transmitted lights have same velocity in all directions (SCOTT 1991). On the contrary, polarised light pictures show the microstructures and grain orientation in the case of the anisotropic alloys. The direction and velocity of the reflected or transmitted lights is dissimilar after interacting with these materials. Even, some metallic materials with high anisotropy can present their microstructure non-etching via this method, e.g. antimony, magnesium, cadmium, cobalt, titanium and tin (McCrone, McCrone, and Delly 1978; SCOTT 1991; Calvo, Gautier, and Simon 1991). Polarised-light pictures from the anisotropic alloys are in grey scale due to distinct reduction of the light velocity that is produced by sample.

In the case of grain orientation, it is observable at different colours because the different degrees of ellipticity. Polarised light can evaluate the grain of the alloy quantitatively and qualitatively via this colour difference. However, specific software is necessary to carry out these analyses (Calvo, Gautier, and Simon 1991). Chemical and electrochemical polishing are used to prepare these type of samples due to the mechanical polishing can produce deformation twins, which can generate errors in the interpretation of the microstructures of alloys (Vander Voort 2007).

Other characteristics of the metallic materials can be analysed through this method, e.g. second phase, refraction index, internal stress, thickness or dispersion. Isotropic second phases usually are viewed like black structures (McLaughlin 1977). Etched samples for polarised light method exhibit low reflection index. For this reason, some cases, samples are commonly inked to increase the contrast of the microstructural elements. Figure 7 shows examples of

the microstructures pictures that can be developed via polarised light OM.

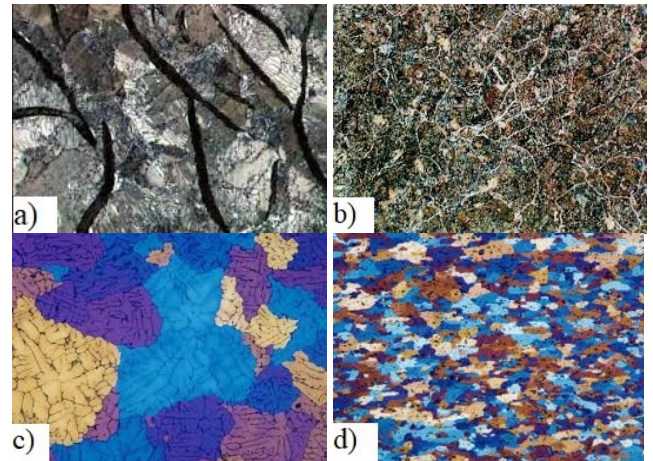


Figure 7: Polarised light OM pictures of a) gray iron, b) eutectic Fe-C (ÇARBOĞA 2014), c) 1100 and d) 2024 aluminium alloy (optics 2018).

It is important to comment that the grains, even matrix, and second phases have various crystalline structures, as such α , β , γ , S, θ , martensitic, dendritic, etc... The objective of the section is not discussing in the structural properties, but discussing the microscopy methods. Although OM is a great technique to evaluate of the metallic alloy microstructures, it is inefficient to analyse structures at nanometres.

4. Scanning Electron Microscopy

Scanning Electron microscopy is a surface analysis technique utilises a focused electron beam for obtaining the pictures of the samples. Electrons beam interaction with the atoms of the samples generates diverse signals, which are interpreted to produce the pictures (Zhou et al. 2006). Manfred von Ardenne is considerate the inventor of the first SEM in 1937. However, the first commercial SEM was fabricated by Cambridge Scientific Instrument Company in 1965 by name of "Stereoscan" and was delivered to DuPont (McMullan 1995; Nada 2015). SEM is a great technique for the metallographic analysis of the metallic alloy because it allows evaluating the topography and chemical composition of the samples at nano-scale level. The utilisation of the electrons in analysis of the samples permits to evaluate the surface features of the metallic material at high magnifications (nanometre). Various signals created by electrons-atoms interactions, which are able to deliver information about the microstructure and chemical composition of the alloys. Some of these signals are difference between the energies of the electrons before and after interacting the samples. One or other type of SEM analysis is depending on these energy variations (Vernon-Parry 2000). Electron microscope is the device utilised to

carry out SEM analyses and therefore, characteristics of this device determine the type of information that can be obtained on metallic alloys. The next subsections therefore describe the features of the electron microscope, Secondary Electrons (SE), Back Scattered Electrons (BSE) and applications on the metallography.

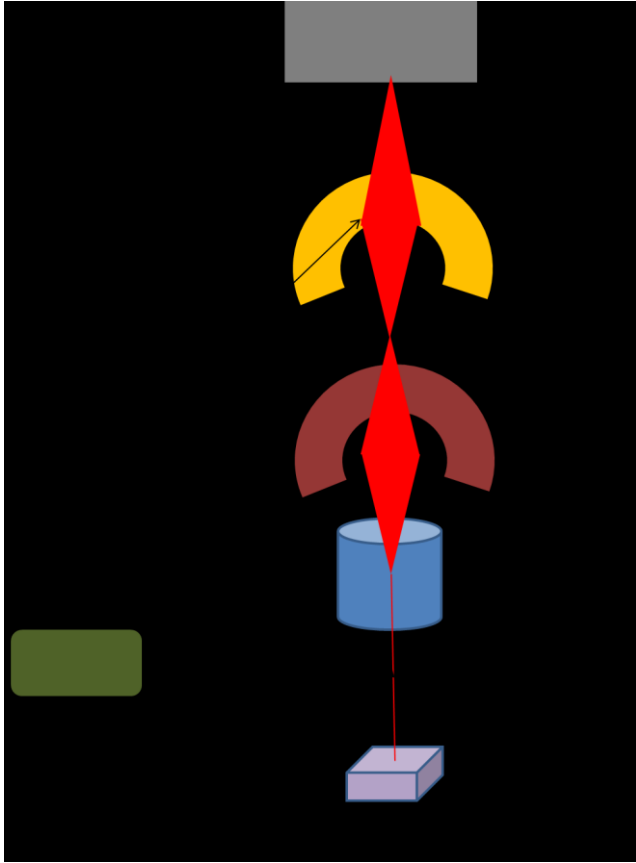


Figure 8: Schematic drawing of the electron microscope.

4.1. Electron Microscope

This facility uses electrons as illumination sources and therefore, its resolution is higher than the OM, which uses light. Electron microscope can be diverse types, but only scanning electron microscope is described in this chapter because it is most widely utilised device in metallographic field. Scanning electron microscope is formed of several elements; electron gun, condenser lenses, deflection coils, final lens, vacuum chamber, amplifier and detector (Ishikawa 1989; Hafner 2007). Figure 8 illustrates the parts of the electron microscope.

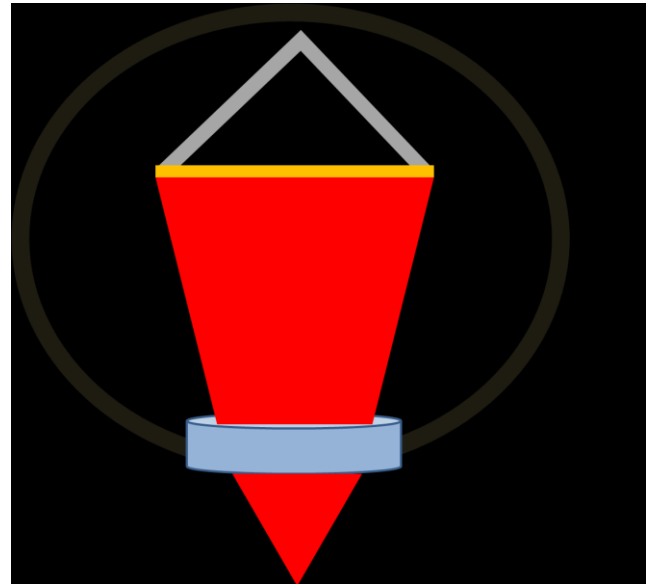


Figure 9: Schematic drawing of the electron gun.

Electron gun is the device that produces the electron beam and it is commonly placed to top of electron microscope (See Figure 9). This device generates a source of electrons and accelerates them through a potential that is from 0.1KV to 40KV (Reimer 2013; Dunlap and Adaskaveg 1997). Electron gun comprises of a hot wire, Wehnelt cap and anode. The hot wire is surrounded by Wehnelt cap and anode is localised at the bottom of Wehnelt cap. Electrons are produced by the hot wire and they are attracted by anode that has a hole, which allows going out electrons to condenser lenses. Wehnelt cap avoids electrons scape of the electron gun (Nada 2015; Dunlap and Adaskaveg 1997; Joel 2009; Aharinejad and Lametschwandtner 1992). Scheme of the electron gun can be observed in Figure 9. High voltages permit hot wire produces electrons.

According to the technique that is used by hot wire to generate the electrons, electron gun can be classified in two types; thermionic and field emission (Nada 2015). Thermionic emission generates electrons by mean of the heating of hot wire via supplying of the high voltages. LaB_6 crystal is the material usually employed to fabricate the hot wire for thermionic emission (Joel 2009). Field emission is called as cold filament because electrons are produced by electrostatic field that is created by elevated potentials. Wolfram crystal, W, is used as material for the hot wire of field emission. Although W crystal can be also used in the thermionic emission, this material has shorter lifetime than LaB_6 (Dunlap and Adaskaveg 1997). The advantages and disadvantages of two techniques are listed in Table 6.

Condenser lenses control the number of electrons that hit the samples and therefore, these items determine the size of the beam. Lenses produce a magnetic field that permits to define the amount of the electrons, which impact on samples (Zhou et al. 2006). SEM commonly has two condenser lenses that

is situated after the electron gun (Cheney 2007; Postek et al. 1980).

Deflection coil or electro lens is the responsible device to produce a square electron beam on surface of the sample and it is localised above condenser lenses. This device is an electromagnet that modifies the shape of the electrons beam through generation an electromagnetic field (Hafner 2007; Joel 2009).

Final or objective lens allow focalising the electron beam on the sample and it is placed after condenser lenses and before sample. An electromagnetic lens is normally utilised as final lens (Zhou et al. 2006).

Vacuum system generates atmosphere of low pressure, 10^{-3} - 10^{-4} Pa, which permit electrons to hit the samples. Any particles or gas in the atmosphere can interact with electrons and hinder them to reach the samples (Joel 2009). Vacuum system is formed of two pumps; the first pump does the rough evacuation, and second pump reaches higher vacuums. Second pump must begin to operate after first pump achieves its lowest pressures (Johnson 1996). To ignore this recommendation can cause dangerous damages on second pump. This system is localised close to the chamber where is place the samples. The most useful pumps on SEM usually are rotatory, diffusion, scroll, turbo-molecular and ion getter. Rotatory pumps are utilised as first pumps to reach at low level vacuum. The device consists in a rotation vane housed in a cylindrical chamber in an oil bath that absorbs the gases from atmosphere to reduce the environmental pressure (Aharinejad and Lametschwandtner 1992; Krivanek et al. 2008). Diffusion pump is used as second pumps and it is formed of cylindrical chamber with series of vanes. Oil also is the responsible to catch the gases from atmosphere, but in this case, the oil is quickly heat and cool (Danilatos and Robinson 1979; Stokes, Thiel, and Donald 1998; Yoshimura 2008). Mechanical or scrolls pumps comprise of two interleaving scrolls, one is fixed and other orbits eccentrically. It allows compressing the environmental gases. These pumps are commonly utilised as first pumps because it is more ecological than other pumps, free oil. Turbo-molecular pumps can be used as second pumps. These pumps consist a series of pairs of rotor fan blades, which some are statics and others dynamics. Spinning of the dynamic blades permits to extract the gases from the atmosphere (Dunlap and Adaskaveg 1997). Other type of the devices used as second pump is ion getter pump or sputter-ion pump that can achieve the highest vacuums. Strong electrical potential is applied to capture and extracting the gases by ionising them. Solid electrode is utilised to reach this process (JUNLI 2008).

Amplifier is the device that increases the received signal from detector, and it is placed after the detector. Photomultiplier is the device that is utilised as amplifier (Moncrieff and Barker 1978).

Detector identifies the electrons from sample and converts them to electrical signals, currents. This is situated in the sample chamber and close to final lens to obtain the best resolution possible. The types of detector are defined by kind of signals that they can detect (Stokes 2008). The types of outputs obtained via SEM are Auger electrons, SE, BSE, characteristics X-ray, continuum X-ray and cathodoluminescence (Stokes 2008). Penetration depth of these signals is dissimilar depending to type as can be seen in Figure 10.

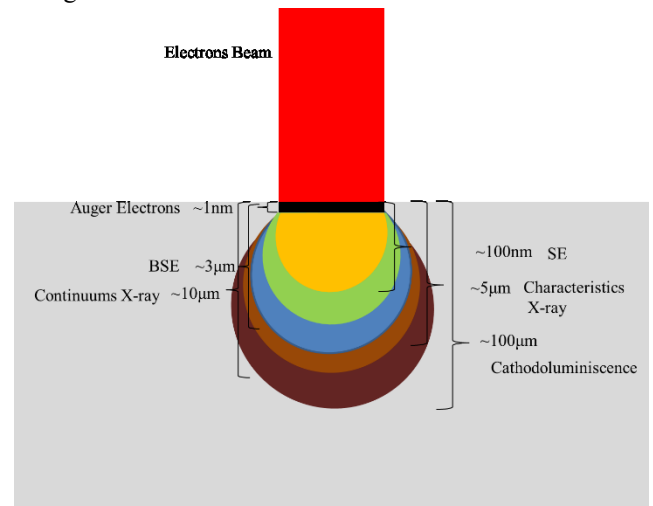


Figure 10: Penetration depth of the SEM signals.

Cylindrical Mirror Analyser (CMA), Hemispherical Deflector Analyser (HDA), Hyperbolic Field Analyser (HFA), Parallel Cylindrical Mirror Analyser (PCMA) is the detector for Auger electrons. CMA has the best resolution whilst HDA can be combined with other detectors. HFA can be also added to other detectors and is fast but, it has the low total transmission efficient. PCMA is similar to CMA though it has a wider energies range (Gunawardane and Arumainayagam 2006; El Gomati 2006).

Everhart-Thornely (ET) and In-Beam (IB) are used to detect SE. ET is placed in the chamber while IB is localised in the final lens. IB provides the best possible resolution and the highest magnifications whilst ET has a high efficient and small noise (Cantoni and Holzer 2014; Hafner 2007). BSE is detected by mean of a solid state semiconductor that is situated in the final lens or IB (Cantoni and Holzer 2014; Goldstein 2003). ET can detect both types of the signals (Goldstein 2003).

Characteristics X-ray are detected by a semiconductor protected via tube. Semiconductors commonly are silicon crystal and coated lithium with gold nano-layer. Protective tube is beryllium window.

Continuum X-ray detector usually is Si(Li) cooled with liquid nitrogen though he silicon drift with Peltier cooling system is currently starting to use due to its rapid data acquisition. These detectors can be also utilised in the

analyses of characteristics X-ray (Goldstein 2003; Goodhew 2001).

Panchromatic and Rainbow detectors are utilised to detect the cathodoluminescence. Rainbow is able to evaluate different wavelength (350 nm-850nm) at the same time (*CL Detectors*), while Panchromatic carries out the analyses of the cathodoluminescence in limited wavelengths ranges (Hamers and Drury 2011).

The mostly used SEM signals in the surfaces analysis of the metallic materials microstructures are SE and BSE. Although the other outputs are utilised to evaluate the characteristics of the alloys, these signals are used to analyse the main chemical composition of alloys or crystalline orientation.

4.2. Secondary Electrons

SE permit to achieve topographic pictures of the material surfaces via inelastic scattered electrons. These electrons are originated by the interaction of the electrons beam with valence electrons of the sample's atoms. Electrons from beam replace the valence electrons from surface atoms, which are ejected from samples to detector (Zhou et al. 2006; Vernon-Parry 2000; Stokes 2008; Goldstein 2003). These electrons are free any influence from atomic nucleus and their penetration into the samples only is 0.5-50nm (Cheney 2007; Stokes 2008). Thus, SEM with SE provides topographic information of the samples.

Topographic pictures are in white and black that are defined by the kinetic energy of the ejected electrons. Light areas are produced by electrons with high energy whilst low energy electrons generate the dark zones in the pictures. Energy of the ejected electrons is determined by the distance between the surface and the detector because electrons go out from surfaces without any influences. The longer distances between surface and detector cause lower energy electrons. Thus, light zones therefore indicate high areas while the dark zones are deep areas (Zhou et al. 2006). Figure 11 shows examples of the SEM imagines with SE.

Nevertheless, determined surface geometries and certain physicochemical properties of the materials can produce erroneous contrast in SEM pictures via SE. The sharp edge on the surface can generate brighter zones than normal.

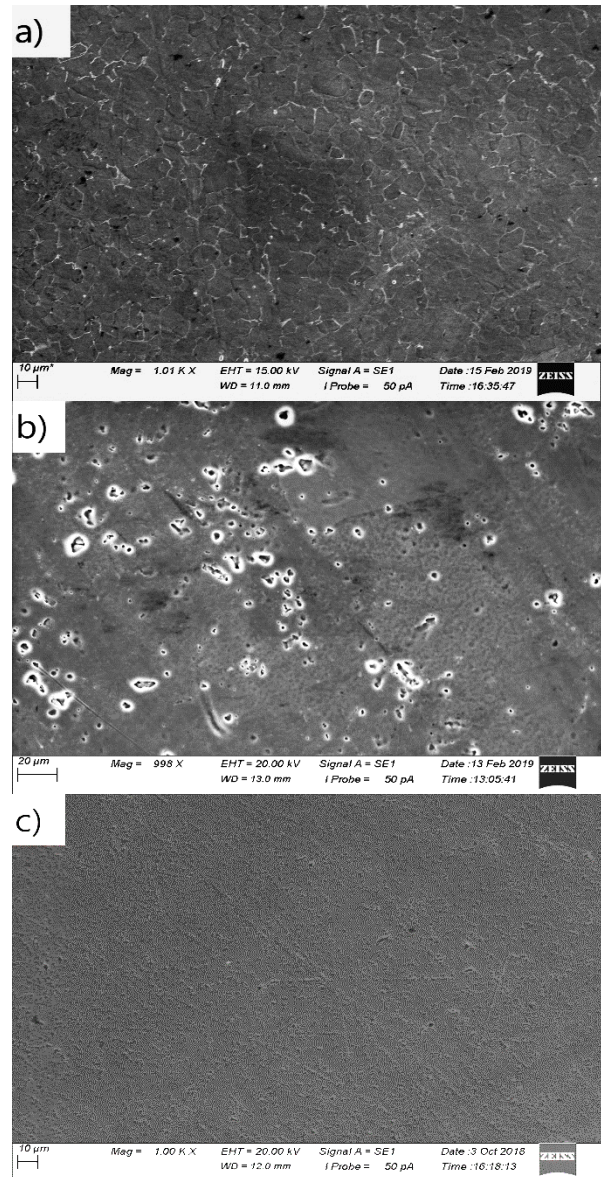


Figure 11: SEM pictures with SE at 1000X magnifications of a) 235 steel, b) 6061 aluminium alloy and c) 430 stainless steel. Stainless steel and steel were etched by 88 etching dissolution according to ASTM E407-99 (E407-07 2015). Aluminium alloys were etched by 3 etching dissolution according to ASTM E407-99 (E407-07 2015).

The brightness of the pictures is proportional to the electrons beam enters obliquely because it changes the distance from surface to detector. This can be observed in crystalline materials. The change of the incident angle is a correct solution at this problem (Joel 2009; Zhou et al. 2006). The semiconductor materials can present the same problem when the exposition time is long. Electrons beam are accumulated by the semiconductor that ejects them after certain time. This generates that the amount and energy of the electrons will be higher than in the real cases (Heydenreich 1993). Short expose time to electron beam is recommendable for this type of samples to avoid this problem. The no electrically conductive materials can cause fake deep areas because the

surface absorbs electrons beams. This produces a reduction in the number of electrons that arrive to the detector. These samples must be coated with conductive layer for resolving this problem (Jbara et al. 2004; Goldstein 2003).

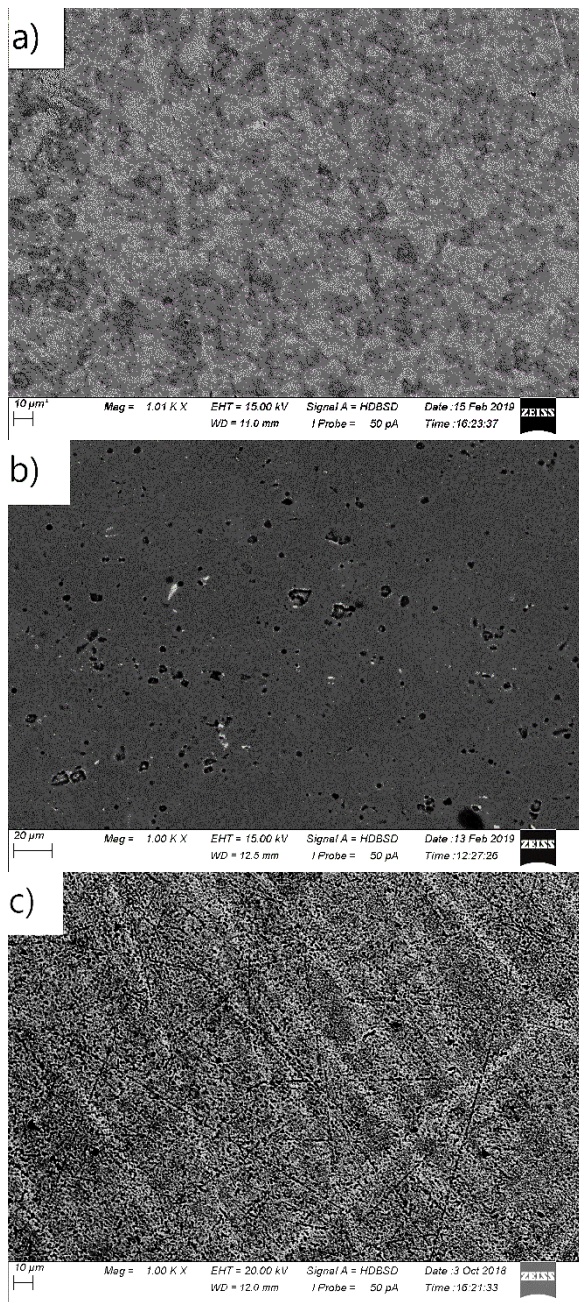


Figure 12: SEM pictures with BSE at 1000X of a) 235 steel, b) 6061 aluminium alloy and c) 430 stainless steel. Stainless steel and steel were etched by 88 etching dissolution according to ASTM E407-15 (E407-07 2015). Aluminium alloys were etched by 3 etching dissolution according to ASTM E407-15 (E407-07 2015).

SEM pictures through SE should be carried out at low accelerating potential, 1-10KV, because the penetration depth of the electron beam is proportional at potential. Characteristics form bulk material can influence in the SEM

pictures at high accelerating potential (Stokes 2008; Aurtata and Hejna 1991)

4.3. Back-Scattered Electrons

BSE create pictures of the semi-qualitative chemical composition through elastic scattered electrons. These electrons are generated by the interaction between electrons beam with the atomic nucleus. Beam electrons have sufficient energy to pass close to the atomic nucleuses of the samples. Then, they leave from samples and arrive to detector (Hafner 2007; Stokes 2008; Goldstein 2003). Electrons beam are modified by crossing close to atomic nucleus, which allow creating SEM pictures with BSE. BSE are therefore originated by electrons beam as opposite to SE. Depth range of the BSE is defined by acceleration potential and it is from 1µm to 3µm (Radetic 2011). For this reasons, SEM via BSE provide chemical composition information. An example of the SEM images carried out with BSE, can be viewed in Figure 12.

As SEM pictures via SE, SEM images with BSE are in grey scale. Light zones also indicate electrons with high energy while low energy electrons are seen as dark areas. High energy BSE is produced when atomic nucleus with high atomic number (Z) interacts with them while low Z atomic nucleus generates BSE with low energies. This is due to the interaction between electrons beam and atomic nucleus is similar than elastic collision. Thus, light areas are zones with high Z elements whilst low Z elements cause the dark areas (Robinson 1980; Joel 2009; Goldstein 2003). Chemical composition of the samples can be therefore analysed semi-quantitatively through SEM pictures obtained by BSE.

Although this technique provides great and fast information on the chemical composition on the samples, SEM with BSE presents some limitations. The topography of the samples modifies the SEM pictures via BSE because changing the distance between sample surface and the detector. As commented in the Secondary Electrons subsection, energies of the electrons are inversely proportional to the surface-detector distance. Peaks of the surface can therefore produce lighter zones than in normal conditions, while deep areas are able to generate darker zones than in regular situations (Joel 2009; Robinson 1980). Polished surfaces are recommendable to conduct these analyses. If it is impossible, it is recommendable to utilise other types of chemical composition analysis techniques for discharging possible errors. SEM of BSE has the same problems than SEM via SE with semiconductor and non-conductive materials. Semiconductors produce fake light zones while the non-conductive materials cause false dark areas. Other problem is generated at high acceleration potential. The depth penetration of the electrons beam is defined by accelerating voltage. An excess of potential can causes that some BSE is

originated from into bulk and not form surface. It can produce erroneous hues in SEM images with BSE. For this reason, moderate potential, $>50\text{eV}$, should be used to carry out the SEM analyses with BSE (Richards, Owen, and Ap Gwynn 1999; Zhou et al. 2006).

4.4. Metallographic Application of the Scanning Electronic Microscopy

SEM is adequate technique to metallographic analysis of the metallic materials because its advantages, as high resolution (nanometers scale), topographic and chemical composition analysis viable. Moreover, metallic materials are recommendable for these types of analyses because they are free coating, conductive and non-semiconductor samples. Metallic alloys are conductive materials and therefore, the gold coating is unnecessary (Goldstein 2003; Johnson 1972). These materials usually are free of the semi-conductive effects. However, certain oxidised metals present semi-conductive properties, e.g. ZnO , Fe_2O_3 or TiO_2 . Thus, the elimination of any oxidation on the metallic materials is very important to carry out an appropriate SEM analysis (Vander Voort 1986).

Note, it is important to highlight that the SEM pictures can be dissimilar than OM because the SEM uses electrons to make the images while the OM utilises light. The interaction of the light with the metallic alloys is totally different than in the case of the electrons. In additions, SEM pictures with BSE are different than with SE due to the detected electrons are distinct origin. For this reason, BSE should use to different aim than the SE. These differences can be seen in the SEM pictures through SE and BSE from Figure 13.

SE are adequate to evaluate the microstructures of the metallic materials as grain, dendritic and martensitic structures. Commonly, grains have lighter tones than borders grain. This is due to the border grain commonly are deeper than the surface because these are preferential zones of attack for etching dissolutions (Vander Voort 1986; AOYAMA, NAGOSHI, and SATO 2017). It is important to remember that SEM with SE generates topographic pictures of the metallic samples. In addition, the geometries of these microstructures can be viewed with better definition than BSE because SE generally is free chemical composition interactions from alloy elements. Nevertheless, intermetallic and second phase can be difficultly to identify due to etching can non-modify the surface topography around these elements.

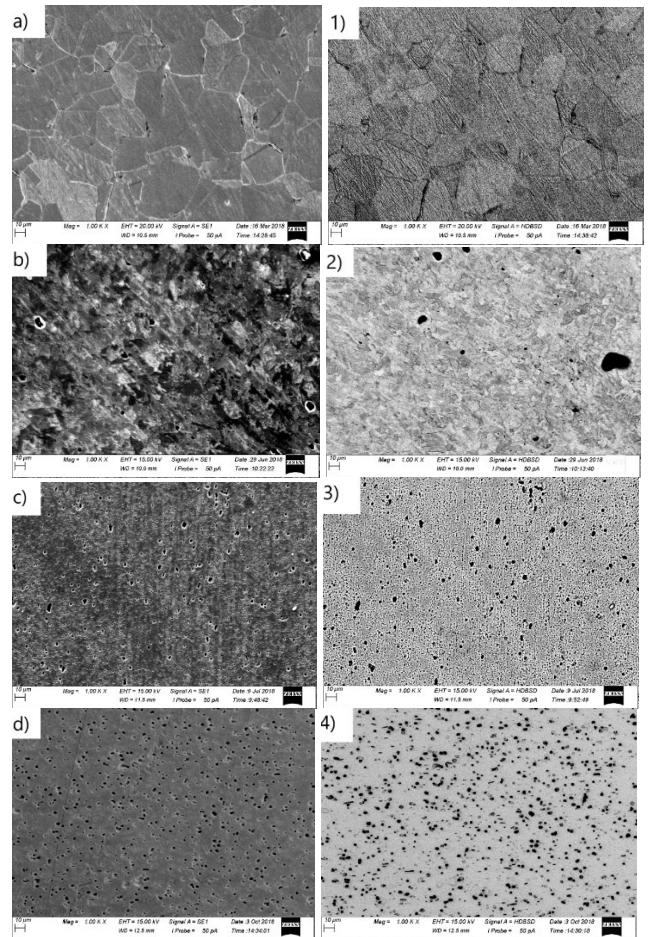


Figure 13: SEM pictures at 1000X by mean of SE (a-c) and BSE (1-4) of a,1) 316L stainless steel, b-2) 355 steel, c-3) 5052 and d-4) 3103 aluminium alloy. Stainless steel and steel were etched by 88 etching dissolution according to ASTM E407-15 (E407-07 2015). Aluminium alloys were etched by 3 etching dissolution according to ASTM E407-15 (E407-07 2015).

BSE are very appropriate to evaluate second phases and inter-metallics. This is due to inter-metallic and second phase commonly have a different chemical composition than metallic material matrix. These microstructures are easily identified by mean of SEM via BSE because its images are about the chemical compositions (AOYAMA, NAGOSHI, and SATO 2017; Lloyd 1987). In addition, the identification of the different types of second phase and intermetallic is relatively easy due to the dissimilar hues that are produced by distinct chemical compositions of these microstructures. Moreover, dispersed elements can be determined by this technique, as long as they have a dissimilar chemical composition than the matrix. It is important to note that the matrix hues can be different according to the Z of its elements. Although this is non-common, grains microstructures can be observable by mean of this technique. This occurs when the borders of the grains have a high depth.

Table 4: Examples of the etching (E407-07 2015; Zipperian 2011).

Metallic alloy	Type of etching	Etching conditions	Developed microstructures
Aluminium alloy	Chemical	<ul style="list-style-type: none"> • 1 mL HF and 200mL water. • 15s • Non-immersion 	General structure
	Electrochemical	<ul style="list-style-type: none"> • 25mg/mL HBF₄ • Al cathode • 30 V DC • 1min 	Grain orientation
	Chemical	<ul style="list-style-type: none"> • 33.33% (mL/mL) HNO₃ • 40s • 343K 	Phases
Copper alloy	Chemical	<ul style="list-style-type: none"> • 20mg/mL K₂Cr₂O₇ • 8% (mL/mL) H₂SO₄ 	General structure
Gold alloy	Chemical	<ul style="list-style-type: none"> • 60mL HCl and 40mL HNO₃ • 2s-120s 	General structure
Steel	Chemical	<ul style="list-style-type: none"> • 5mL HNO₃ and 100mL ethanol or methanol • 2s to 60s 	General structure
Stainless Steel	Chemical	<ul style="list-style-type: none"> • 5mL HCl, 1g picric acid and 100mL ethanol or methanol • 15min 	General structure
	Electrochemical	<ul style="list-style-type: none"> • 1.7g/mL NaOH • 6V • 5-10s 	Stains sigma phase
	Electrochemical	<ul style="list-style-type: none"> • NH₄OH concentrated • 6V • 30-60s 	Carbides and sensitization
	Electrochemical	<ul style="list-style-type: none"> • 0.2g/mL NaOH • Stainless Steel Cathode • 2-20V DC • 5-20s 	Delta ferrite
Lead	Chemical	<ul style="list-style-type: none"> • 15mL Acetic acid, 15mL HNO₃ and 60mL glycerol • 353K 	General structure
Platinum	Chemical	<ul style="list-style-type: none"> • 5mL HNO₃, 25mL HCl and 30mL water • 1-5min 	General Structure
Magnesium alloy	Chemical	<ul style="list-style-type: none"> • 1mL HNO₃, 20mL acetic acid, 60mL diethylene glycol and 20mL water • Non-immersion • 1s-3min 	General Structure
	Chemical	<ul style="list-style-type: none"> • 10mL Acetic acid, 6g picric acid and 100mL ethanol or methanol • 15-30s 	Phase
Silver	Chemical	<ul style="list-style-type: none"> • 50mLNH₄OH and 20mL H₂O₂ • 2s-60s 	General structure
Titanium	Chemical	<ul style="list-style-type: none"> • 10mL HF, 5mL HNO₃ and 85mL water • Non-immersion • 2-20s 	General structure

Table 5: Summary of other optical microscopy techniques.

Technique	Light	Modification	Main application
Stereo microscopy (Schreier, Garcia, and Sutton 2004)	Reflected	Light source out of microscopic structure	Biomedical and industrial
Comparison microscopy (Wilkinson 1954)	Transmitted	Use two microscope connected by eyepiece	Forensic
Inverted microscopy (Liu, Lu, and Sun 2010)	Transmitted	Source light and condenser are placed on the top while objective lenses and eyepieces are localised on the bottom	Medical and biological
Travelling microscopy (Manoharan and Lewandowski 1989)	Transmitted	Rail to move the microscope	Big samples
Fluorescence microscopy (Wayne 2019)	Phosphorescent or fluorescent	Excitation filter, dichroic mirror and emission filter	Biomedical
Confocal microscopy (Semwogerere and Weeks 2005)	Transmitted or laser	Spatial filter and possible laser	Crystallographic and biomedical
Two-photon excitation microscopy (Svoboda and Yasuda 2006)	Fluorescent	Near infrared laser, red filter, green filter and dichroic beam splitter	Biomedical and chemical
Ultramicroscopy (Dodt et al. 2015)	Scattered	Convergent beam	Nanoparticles and colloids

Table 6: Advantages and disadvantages of the thermionic and field emission (Dunlap and Adaskaveg 1997; Goldstein 2003).

Technique	Advantages	Disadvantages
Thermionic emission	High beam current stability	Short lifetime
	Low stringent vacuum	
	Non-period emission flash	
Field emission	Small size of electron source	Inadequate for energy dispersive spectroscopy technique.
	Long lifetime	
	High resolution	
	High magnification	
	Low energy spread	
	High Brightness	

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