41685

Materials Characterisation and Testing

Team 3 - Final Project

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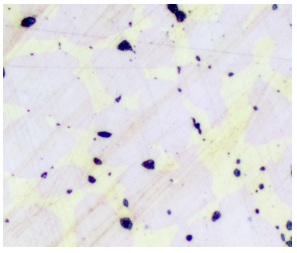
Contents

Introduction

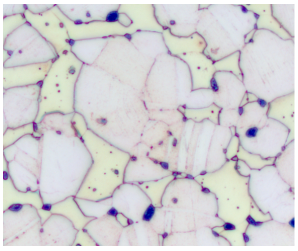
At the beginning of this project, each team was given a sample of a material with the goal of testing it and identifying which material it is. Using different techniques learnt throughout the course, this task was carried out.
Our team obtained a sample that, at first sight and to judge by its colour, looked like copper. However, its colour was a mixed of the typical red of copper, and yellow. Therefore, it was probably a copper alloy.
In the following sections, the different test techniques are going to be presented, together with the results obtained.

Light Microscopy

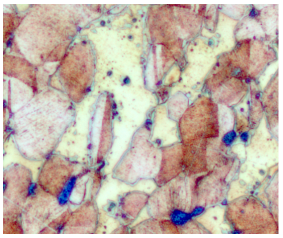
Reflected Light Microscopy (RLM) is often referred to as incident light, epi-illumination, or metallurgical microscopy, and is the method of choice for fluorescence and for imaging specimens that remain opaque even when ground to a thickness of 30 microns. The range of specimens falling into this category is enormous and includes most metals, ores, ceramics, many polymers, in between many more. [1] Therefore, this technique was the first one used to approach our sample. It was observed that it was composed of 3 main components: big red/brown grains, small black spots and a yellow background (see Figure 1c).



(a) Polished cross-section



(b) Rough cross-section



(c) Longitudinal-section

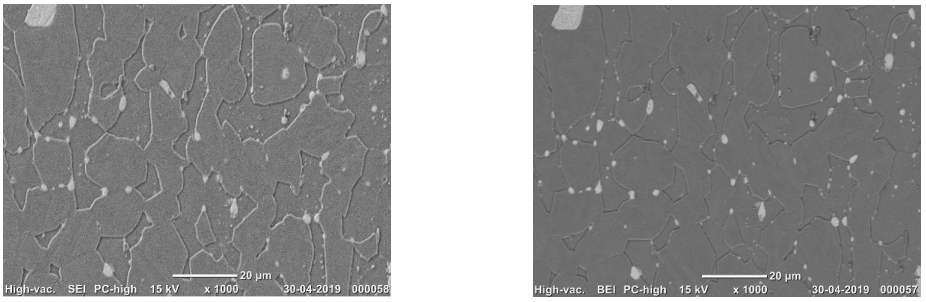
Figure 1: Images obtained using RLM, magnification x100.

After some research, we got to the conclusion that the yellow factor could be due to the presence of zinc. This way, the sample could be the copper and zinc alloy called *brass*. However, this hypothesis needed to be tested in more depth. Hence, more tests were made with Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD).

Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) is a technique used to produce images of a sample by scanning the surface with a focused beam of electrons.[2] There are two different types of SEM imaging:

- Secondary Electrons (SEI): it offers images with resolution independent of the material, obtaining its topographical information.[3] In Figure 2a, it can be seen the sample's topography gotten out of the SEI imaging, where grains can be seen together with a flatter background, and lighter spots of another composition.
- Backscattered-Electron (BSE): it detects elastically scattered electrons,which are higher in energy from atoms below the sample surface.[3] The contrast using this method depends on the atomic number of the components in the sample, so components with higher Z are shown in brighter colour. In this case, spots of a brighter colour can be seen over a darker background. Hence, we can expect to get at least two different components; probably a copper alloy with traces of a heavier element. See Figure 2b.



(a) SEI imaging. (b) BSE imaging.

Figure 2: Image showing the results

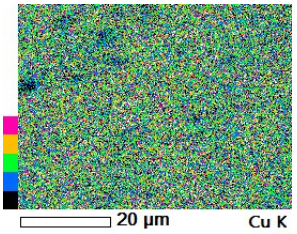
Hardness measurements

As mentioned above, the brighter grains seen in Figure 2 means that there is a heavier component in the sample, in form of grains. The question now is, how much heavier are these grains in comparison to the rest? Willing to investigate this hardness difference between the components, indentation hardness measurements were carried out.
However, during these tests we encountered our first problem. The grains of the heavier compound were far too small to be tested using this method. Therefore, only the hardness of the copper-zinc alloy was measured, as well as the overall hardness. When a load of 1g was used, the big red grains obtained a hardness average value of 126.95; and the yellow-like background, 311.
When the load was increased by a factor of 10^4, meaning that now it was 10 kg, the overall

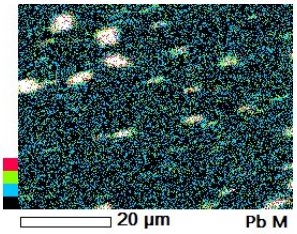
hardness was found to be 132.
This test technique, in our case, did not give us information necessary to find the composition of the sample. However, it was an interesting experience to work with such precise machines, and learn from its sophisticated mechanism.

Energy Dispersive X-Ray Spectroscopy

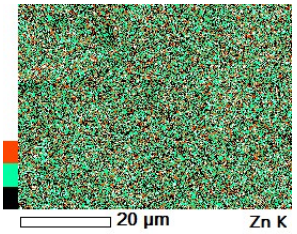
By using Energy Dispersive X-Ray Spectroscopy (EDS) with BSE imaging, the sample was analysed. It was found to be made of copper (60.21%), zinc (28.23%) and lead (10.39%). To see how these components were distributed around the sample, the image was mapped. Below, the mapped images are shown.



(a) Copper mapping.

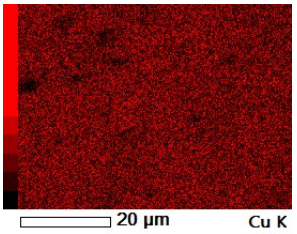


(b) Lead mapping.

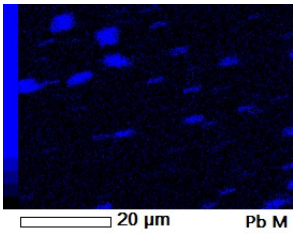


(c) Zinc mapping.

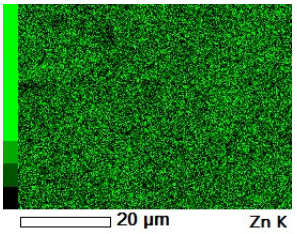
Figure 3: Mapping images



(a) Copper overlay mapping.



(b) Lead overlay mapping.



(c) Zinc overlay mapping.

Figure 4: Overlay mapping images

When overlaying all the mapped images together to see the sample as a whole composition of Cu, Zn and Pb, it can be observed the accuracy of this method. See Figure 5.

From this image, it is clear that there is a background composed of copper and zinc, with grains of lead in the composition, as it was foretold at the beginning.

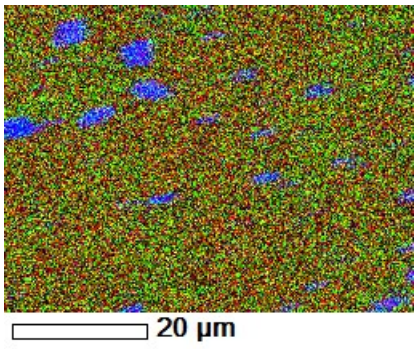


Figure 5: Overall overlay of sample.

X-ray Diffraction

X-ray crystallography (XRC) is a technique used for determining the atomic and molecular structure of a crystal, in which the crystalline structure causes a beam of incident X-rays to diffract into many specific directions.[4] By measuring the angles and intensities of these diffracted beams, a crystallographer can produce a three-dimensional picture of the density of electrons within the crystal. From this electron density, the mean positions of the atoms in the crystal can be determined, as well as their chemical bonds, their crystallographic disorder, and various other information.
Putting into mathematics form, it's Bragg equation [5]:

$$2d\sin(\theta) = n\lambda$$

where d is the distance between lattice layers, θ is the angle, λ is the wave length.

Following Bragg's law, a difractorgram is shown in Figure 6.

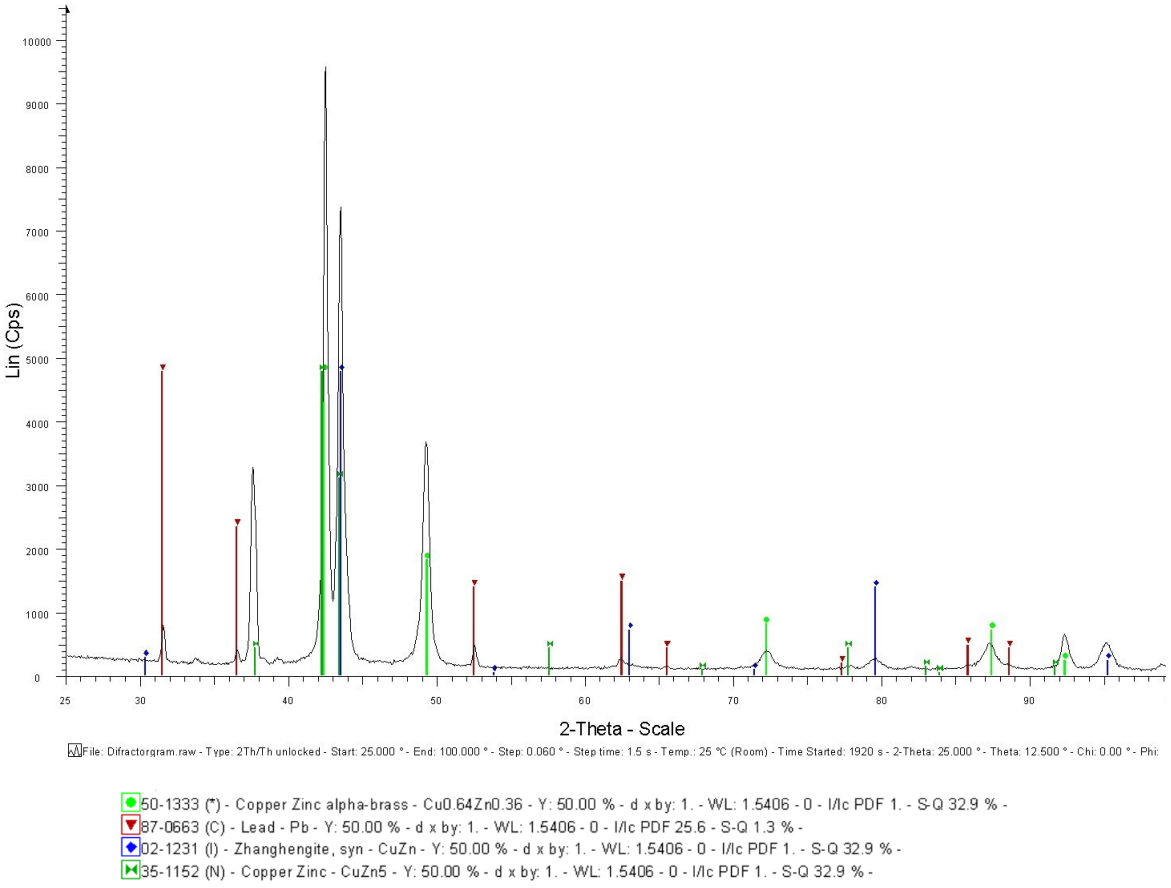


Figure 6: Difractorgram and grain structure

As it can be observed in the legend of the diagram showed in Figure 6, the composition of the material structure is BCC and FCC combining with mixes of both. α and β phases both presents in this material, this is an $\alpha + \beta$ phase structure.

Phase diagram and micro-structure

Now that it was obtained a specific brass type, alpha-beta brass, it is time to see if this conclusion makes sense. To do so, the phase diagram of brass is going to be analysed. It can be seen in Figure 7

From this diagram, it can be observed that brass has mainly 4 phases with different characteristics: $\alpha, \alpha+\beta, \beta, \beta+\gamma$ and γ; depending on the temperature and zinc concentration. According to the percentages of copper and zinc found using EDS (60.21% Cu;28.23%

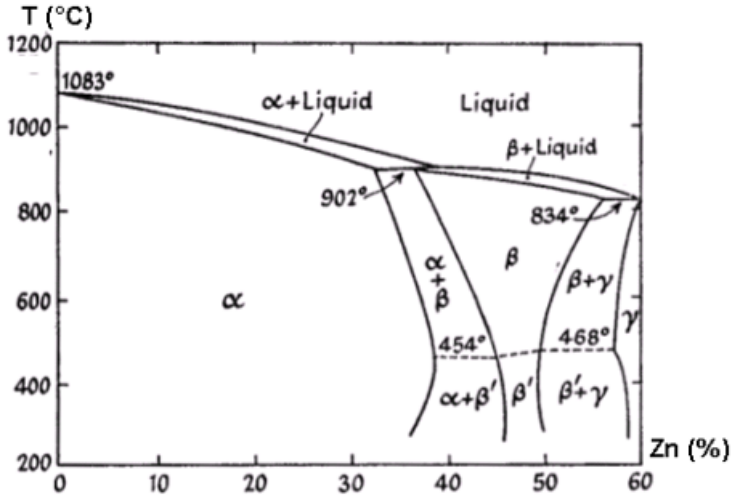


Figure 7: Phase Diagram of brass.

Zn), the classification for this sample is Alpha-beta brass, which percentages oscillates in between 55-65% Cu and 35-45% Zn. Therefore, the theory has matched the results used obtained in XRD, Figure 6.
These type of brasses are also called *duplex brasses*, and are suited for hot working. They contain both α and β'-phases, being β'-phase body-centered cubic (BCC), which is harder and stronger than α. As there is a high percentage of zinc, it means that these brasses are brighter than alpha brasses. [7].

Conclusion

After obtaining all the results from these techniques and comparing them, it was confirmed that the sample was, indeed, the copper-zinc alloy *brass*. Moreover, the darker spots seeing in the Light Microscopy were found to be traces of lead, which is heavier and does not dissolve in water.
After analysing the phase diagram of brass, and comparing it to the experimental results obtained by XRD, it was concluded that the sample is of the type alpha-beta brass.
Moreover, comparing the results found with the different techniques and Figure 8, it can be concluded that the material tested is C36000 Free machining brass [6].

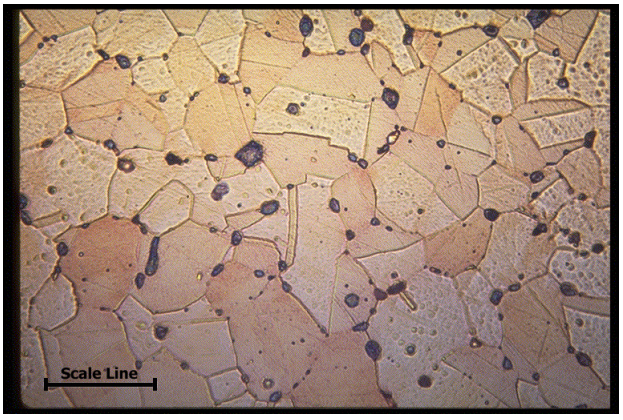


Figure 8: C36000 Free machining brass [6]

Work Distribution

Task \ Name	Andrea Boa	Ku Zhu
Overall experiments	50	50
RLM	100	0
SEM	50	50
XRD	0	100
Conclusion	50	50

Bibliography

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[4] Wikipedia - *X-Ray Diffraction*: https://en.wikipedia.org/wiki/X-ray_crystallography

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