VOL. 11, NO. 16, AUGUST 2016 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences


www.arpnjournals.com

CASE STUDY: TECHNIQUES FOR MATERIAL CHARACTERISATION OF BRASS TENSILE TESTING FRACTURE SAMPLE

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ABSTRACT

This paper characterised a fracture brass sample that underwent tensile testing. The aim of the case study is to characterized the brass sample with unknown microstructural arrangement and unknown elements composition. Tests were conducted based on the SEM imaging technique, EDX chemical analysis and XRD microstructural spatial analysis to indentify and correlate evidence on the sample. Examinations by morphological SEM analysis of tensile fracture surface shows that the brass sample exhibit a typical ductile like material fracture. XRD analysis confirmed the crystal arrangement of the brass sample is of a single FCC α-phase crystal arrangement. The brass composition is confirmed as 63Cu:37Zn wt% from the test conducted by the XRD analysis. The sample is suspected to undergone annealing for process history from the evidence on twinning on the cross-sectional SEM imaging. Thus it can be confirmed that material is a brass sample with 63Cu:37Zn wt% elemental composition with single FCC α-phase crystal arrangement.

Keywords: material characterization, brass, fracture.

INTRODUCTION

Mechanical testing is done on a given material to assess its mechanical properties and its deformation behaviour. Few example of commonly use mechanical testing are tensile testing, charpy impact test, four point bending test, fracture toughness test and many more [1]. However the supporting material characterisation analysis is an important aspect on understanding the type and mechanism of failure the material undergo. Not only that, the characterisation will also compliment the understanding of the microstructure arrangement of the material. This in turns will assist on investigation of microstructral deformation which happened during the test.

There are a number of specimen sizes and shapes for laboratory testing in order to measure the characteristic properties and to determine fracture behaviour for ductile metals under quasi-static loadings. In general, the three types of specimens used in the quasi-static loadings of ductile metals are; i) smooth specimen, notched specimen and cracked specimen. The measured fracture property and observed macroscopic failure mode also vary from one type of specimen to the other. Not only that, the size of the specimen such as thickness to width ratio does also effect its fracture property and observed macroscopic failure mode [1]

Brass is copper alloyed with zinc. The microstructure and properties of brass is depends on the content of zinc in the solution. It exhibit good strength and corrosion resistance characteristics. Brass colour changes from a dark reddish brown to a light silvery yellow, as the zinc content is increased. Brasses with zinc content up to approximately 35% zinc are single phase of α-brass. The α-brass has good strength and ductility, and is easily cold worked. Cold working increases brass yield strength but reduces its ductility. A single-phase α-brass with zinc content up to 37% can be obtained with careful control of annealing temperature and cooling rate [2].

Brass containing between 32 and 39% zinc have a two phase structure, consist of α and β phases. Yellow brasses are in this intermediate category of brasses. Brasses containing more than 39% zinc, have a predominantly beta structure. The β phase is harder than the α phase. These materials have high strengths and lower ductility at room temperature than the alloys containing less zinc. The two phase brasses are easy to hot work and machine, but cold formability is limited [2].

In this study the emphasis is focus on the material characterisation of the brass fractured sample using imaging technique, chemical analysis and microstructural spatial analysis. The observed results from the analyses then relate to each other to better understand the material characteristics of the fractured brass sample.

EXPERIMENTAL WORK

A quasi static monotonic tensile testing has been conducted on a thin flat brass of about 1mm thick with nominal composition of 63Cu:37Zn wt%. Imaging analysis has been conducted on i) the as is half-pair of fractured sample and ii) mounted, polished and etched sample.

Imaging of the sample is conducted on Philips XL 30 Scanning Electron Microscope (SEM) with tungsten filament electron source. Secondary electron (SE) images are taken on the side view and plan view of the fractured samples. Analyses conducted on the micrograph images will help on characterising the physical deformation the material undergo during the test.

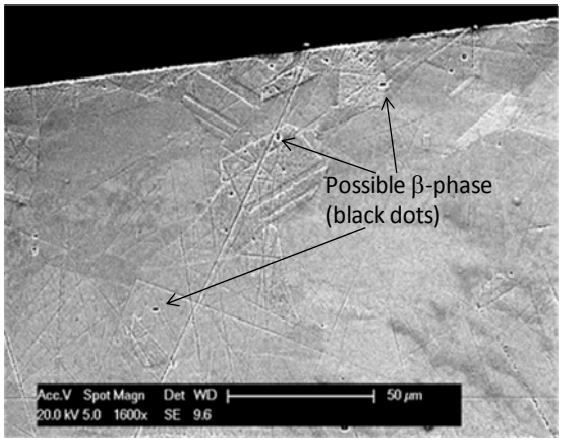
Chemical composition analysis using Energy Dispersive X-ray (EDX) technique is done in the vicinity of SEM with accompanying Oxford Instruments INCA EDX microanalysis system. Compositional quantification is also conducted on pure Cu and pure Zn to act as the

benchmark to the quantification of the brass sample. Microstructural spatial analysis is done by X-Ray Diffractometry (XRD) with Cu-Kα X-ray beam.

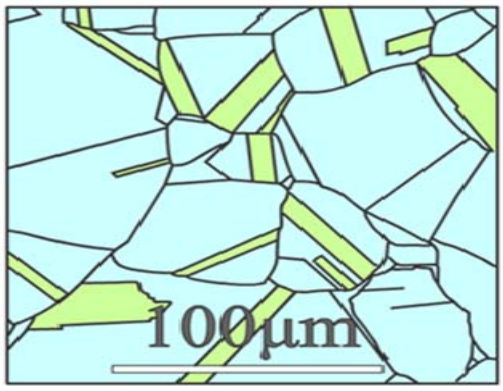
RESULTS AND DISCUSSION

Imaging analysis

Figure-1a shows the Secondary Electron (SE) SEM images of the polished untested specimen. Poor specimen etching prohibits the microstructure to be exposed. There are some grinding marks on the specimen. A schematic crystal orientation of α-brass is shown in Figure 1b. It can be clearly seen the similarity in terms of the geometry features of grain and its orientation suggesting the possibility of single phase α-brass.



(a)

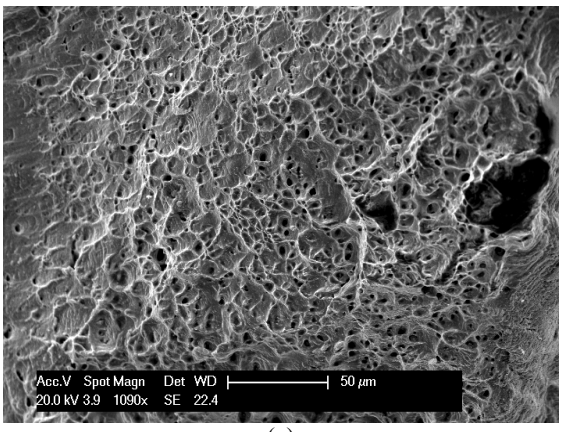


(b)

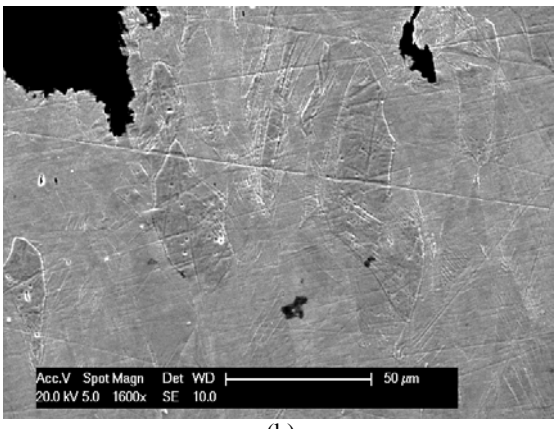
Figure 1. (a) SE-SEM image of the untested polished mounted specimen, and (b) schematic microstructural crystal arrangement for α-brass and annealing twin pattern [3].

There is a relatively small count of tiny black dots believes to be the β–phase of the brass. Another features observed is randomly oriented grains in polycrystalline material suggesting isotropic mechanical properties of the brass sample. Annealing twins is observed in the form of parallel straight line in a grain as seen from the image. This suggesting the brass sample has undergone annealing prior to testing. Further the big grain size suggesting the crystal has been re-growth during the annealing process. This increases the ductility of the brass sample. The grains observed are equiaxed with grain size ranges from about 10 to 50 μm.

Figure-2 shows the Secondary Electron (SE) SEM images of the fractured specimen. The fractograph in Figure-2a is taken on a plane perpendicular to applied tensile loading (i.e. tensile loading is applied on normal axis of this page plane, out of the page). It is also important to note that the specimen on the image is tilted about 30° clockwise on vertical axis of the page. Good depth of field of the image enables two distinct fracture patterns to be observed. In the middle of the sample, it can be seen clearly that there is a huge number of spherical dimples present. While at the side of the specimen, which is in slightly deeper plane, the surface seems to be highly stretch and possibly on some slope.



(a)



(b)

Figure 2. SE-SEM images of (a) as is half-pair of fractured sample, and (b) fractured, mounted and polished sample.

The features observed are similar to a ductile type of failure with plastic straining. Upon yielding and further plastic straining, microvoids starts to appear in the interior of the material. The voids keep enlarging and coalescence to form the crack. As the crack continues to grow and spreads laterally towards the edges of the specimen, the material fails by plastic shearing at maximum shear plane of 45° degree angle due to plane stress effect. This behaviour results a cup-cone fracture pattern normally observed in ductile failure [4]. The fracture mechanism is further confirmed by the mounted, fractured specimen shows elongated crystal grain in the direction of loading suggesting plastic straining (Figure-3).

Figure-3 shows the EDX spectrum obtained from the brass sample showing intensity (counts) of K_α and K_β X-rays of Cu and Zn. Based on the intensity of peak of each element it can be roughly estimates that the composition of Cu to Zn ratio is about 2:1.

Number of K_α X-ray detection counts for a constant period of detection from the brass sample is compared with 3 different methods to quantify the composition of Cu and Zn in the brass. The first method is by comparing counts of K_α X-ray from a standard pure Cu and from a standard pure Zn with brass sample Cu and Zn K_α X-rays. Second method is by comparing Cu and Zn K_α X-rays with brass with known 50wt% Zn.

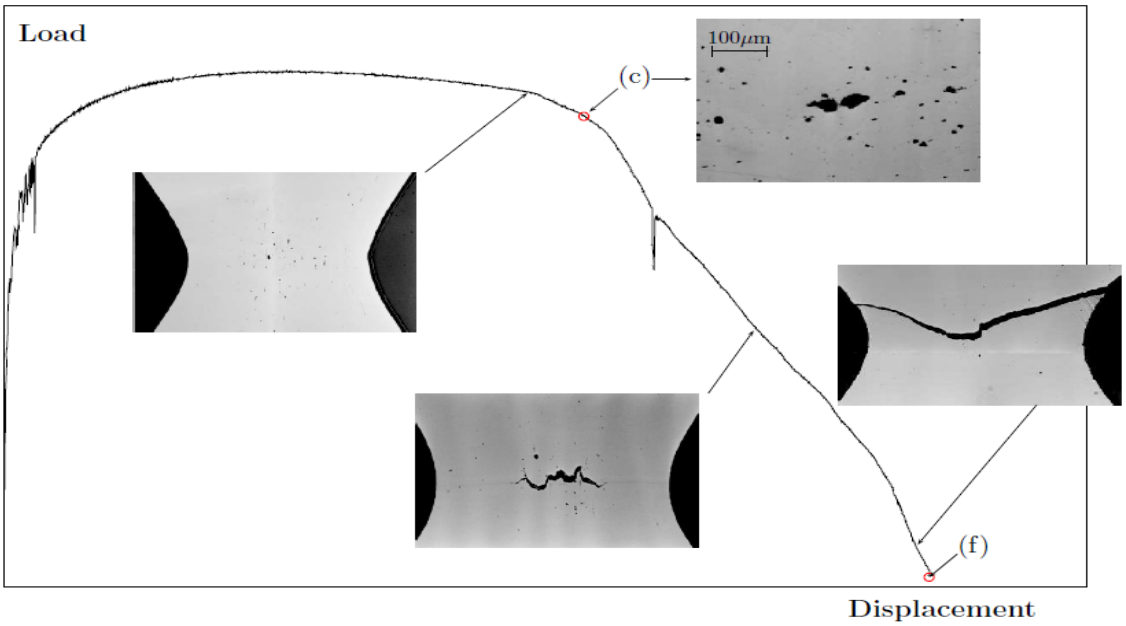


Figure-3. Stages of fracture during tensile testing of specimen, after [4].

Figure-4 shows the EDX spectrum obtained from the brass sample showing intensity (counts) of K□ and K□ X-rays of Cu and Zn. Based on the intensity of peak of each element it can be roughly estimates that the composition of Cu to Zn ratio is about 2:1.

Number of K□ X-ray detection counts for a constant period of detection from the brass sample is compared with 3 different methods to quantify the composition of Cu and Zn in the brass. The first method is by comparing counts of K□ X-ray from a standard pure Cu and from a standard pure Zn with brass sample Cu and Zn K□ X-rays. Second method is by comparing Cu and Zn K□ X-rays with brass with known 50wt% Zn.

The ratio of brass specimen K series counts of Cu and Zn element is divided by the pure Cu and pure Zn standard specimen counts to quantify the brass sample composition based from the pure Cu and pure Zn specimen. For the 50-50 Cu-Zn comparison similar calculation is conducted but the value is then multiplied with 0.5 as the composition on the standard specimen is 50-50. Finally, all the ratios calculated are normalised to get exactly 100% of composition.

The final method is by direct measurement done by the software with ZAF correction applied [5]. Z is the so called atomic number correction – is made up of stopping power and backscatter terms. A is the absorption correction – takes into account that some of the X-rays produced in sample volume don't make it out of the sample. F is the fluorescence correction – corrects for X-ray induced excitation in the sample.

The composition ratio of 62.95% Cu and 37.05% Zn with percentage error of -0.08% and 0.14% are obtained by software for the brass specimen ZAF corrected quantification. The percentage error of the compositional quantifications to the nominal composition of 63Cu:37Zn wt%. are then calculated. The results are shown in Table-1.

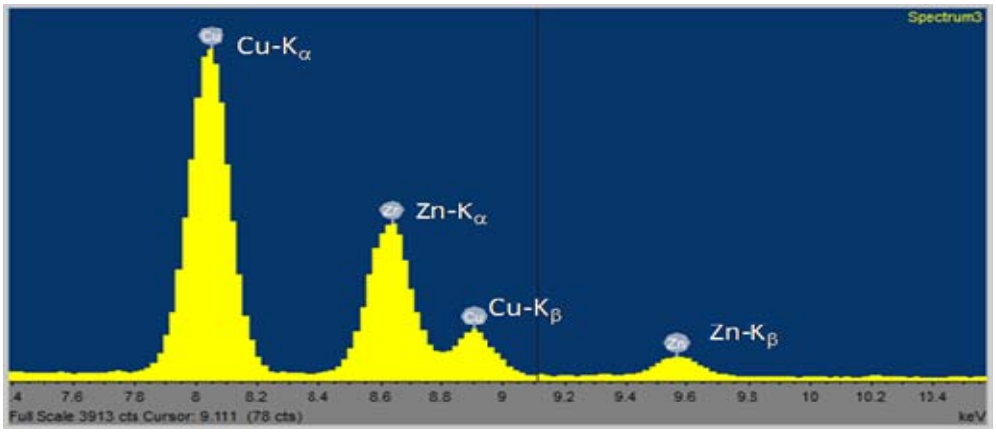


Figure-4. EDX spectrum from brass sample showing K_α and K_β X-ray of Cu and Zn.

Table-1. Quantitative analysis of the chemical composition of the brass specimen.

	K series Net (Counts)		Initial brass/std specimen ratio		normalize brass/std specimen ratio		Real error %	
Element	Cu	Zn	Cu	Zn	Cu	Zn	Cu	Zn
Pure Cu std	58955	-	65.45	-	64.02	-	1.62	-
Pure Zn std	-	50857	-	36.79	-	35.98	-	-2.76
50-50 Cu-Zn std	30987	25322	62.26	36.93	62.77	37.23	-0.37	-0.63
Brass specimen	38586	18705	-	-	62.95	37.05	-0.08	0.14

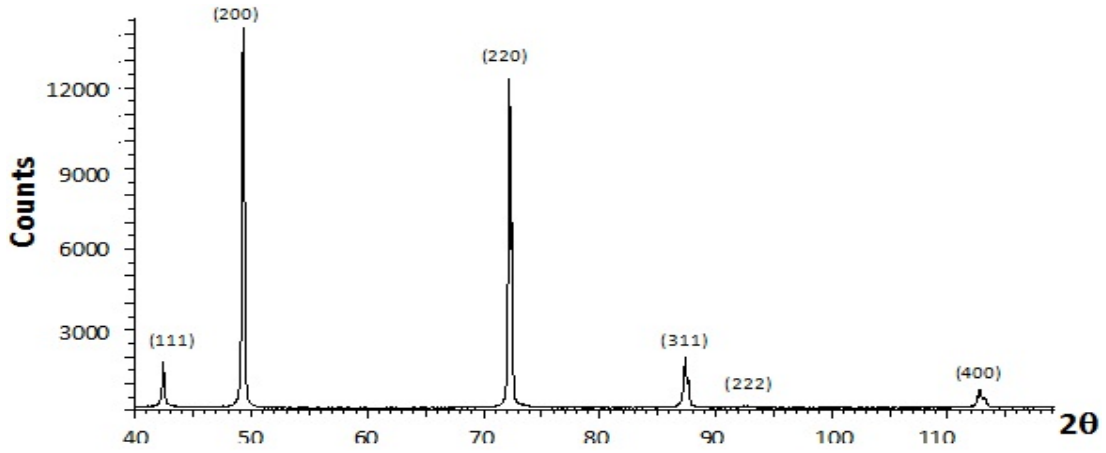


Figure-5. XRD spectrum from the brass sample.

There are slight discrepancies from the 3 different quantitative methods. It can be seen that the most accurate measurement is the one done by the software with ZAF correction being applied. The worst measurement is from pure Cu and Zn standard specimen. This might be due to the slightly high ZAF error which hasn't been corrected for the pure standard specimen.

The XRD spectrum is shows in Figure 5. The intensity of 2:2:1 pattern of peaks shows characteristics of FCC crystal packing for the α-phase brass sample. This confirms the material ductility since FCC crystal arrangement consists lots of plane with high atomic density [6]. There is no evidence of BCC crystal arrangement of the β-phase brass sample. It might not be pickup by X-ray beam since the presence is low [7].

Thus it can be confirmed that the sample is a brass sample with composition of 63wt%Cu and 37wt%Zn

and having an α-phase FCC crystal arrangement. This can be confirmed by tthe ductile behaviour of the material, since FCC arrangement will fail by ductile failure as it has the highest slip plane. Further, the annealing process which cause the twinning in the microstructure is causing the material's ductility to increase.

CONCLUSIONS

Material characterisation case study is conducted on a brass sample that underwent tensile testing. This study is focussing on the technique of imaging through SEM analysis, crystal arrangement by XRD analysis and elemental composition EDX analysis. The work highlighted the failure mechanism and deformation experienced by the sample. Through investigation from imaging technique and characterisation analyses, the following conclusion can be made from the current study;

- The brass sample exhibits a ductile type cup-cone fracture as observed from the fractograph and elongated grains SEM images,
- The composition of the specimen is in agreement with its nominal composition of 63wt%Cu and 37wt%Zn,
- The material is a single phase brass with FCC crystal arrangement which confirms the material ductility
- The sample is suspected to underwent annealing for process history.
- The analyses evidence correlate each other well as annealing and FCC crystal structure will increase the material's ductility. This is confirmed by ductile fracture morphology evidence. The composition found also align with the Cu-Zn phase diagram.

ACKNOWLEDGEMENTS

Authors would like to thank Universiti Tun Hussein Onn Malaysia for providing financial assistance for the project conducted through the grant of RACE 1441.

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