Failure surfaces morphology for specimens with stress concentrators using scanning electron microscopy

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Abstract. Materials develop different type of behaviours based on environmental conditions, loading conditions and different processing methods. Following up a previous work, the authors noticed that a mainly ductile material had features of brittle failure when the material was used for specimens with stress concentrators. Three types of specimens with stress concentrators were chosen and tests were carried out in order to develop a triaxial state of stress using a universal testing machine. After the failure, new specimens of the failure surface were debited and they were studied using SEM analysis. Elements of both brittle and ductile failures were identified and the study went further to verify the chemical composition of the material and the presence of impurities, if any. The authors concluded that the processing methods, the chemical composition and contamination of the material greatly influences the behaviour and type of failure of the specimens.

1. Introduction
In order to study the triaxial state of stress, several methods to develop such stresses are used. They refer to either designed triaxial testing machines, or certain devices that are attached to the universal testing machine or the easiest way by using specimens with stress concentrators. The main function of these stress concentrators is that of obtaining triaxial state of stress under uniaxial loading [1].

Based on the previous work of Malcher and Mamiya [1], the authors have chosen three types of specimens that develop triaxial state of stress, even if the loading is uniaxial. Each specimen has a different type of stress concentrator and have been previously studied from both AEF point of view and the experimental point of view by Comanici et. al [2, 3].

After the uniaxial traction had taken place, new specimens were debited in order to study the morphology of the failure surface.

The material used for these particular specimens is a light non-ferrous metal, being an aluminium alloy called AlMg3 or 5754A, formed by either extrusion, rolling or forging. This type of material is processed at high temperatures, has good mechanical characteristics, high mechanical strength, can be used for welding and is corrosion resistant only if surface-treated or other protections are used [4].

The applicability of this material ranges from aerospace, military equipment to machinery components or rivets and pins. The forms under which this material is sold are wires, rods and bars, but also sheets and boards. There are different classes of aluminium alloys, depending on the use of alloys or their mechanical properties [4].

Their alloying structure varies, but a normal duralumin composition typically consists of an aluminium base with varying percentages of copper, chromium, magnesium and manganese, or even silicon.
Table 1. Chemical composition of 5754 aluminium alloy (wt%).

|   | Al  | Si  | Fe  | Cu  | Mn  | Mg  | Cr  | Zn  | Ti  | Residuals |
|---|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----------|
|   | 94.2 to 97.4 | 0.4 max | 0.1 max | 0.5 max | 2.6 max | 0.3 max | 0.2 max | 0.15 max | 0.15 max |

In order to increase specific properties, the process of obtaining this material is under particular conditions so that the alloy has no impurities. The manufacturing processes are carried out according to the standards in force but microscopic analysis of the material is also required in a section resulting from the breaking with a stressed state, to validate the results.

Since this material is used in the aeronautical industry or to manufacture components of machinery, it is necessary to dispose of a material without impurities, homogeneous and of superior quality.

2. Materials and methods

In the present paper, the authors use an aluminium alloy that has the following concentrations Al 96.5, Mg 3.1, Mn 0.25 and Cr 0.15 (wt.%).

![Figure 1](image1.png)

Figure 1. Test specimens that undergo uniaxial traction (a-Erice, b-Bao, c-Allan).

Following the uniaxial traction tests and the stress-strain diagram analysis, a ductile type of failure was observed for all three types of specimens. When examining the failure surfaces, the presence of ductile cone cups features, as well as brittle-specific elements were found. To confirm these remarks, an electronic microscopy analysis was performed.

The samples (figure 2) were cut by milling as follows: on the flat specimens were isolated portions with surfaces of about 1 cm², adjacent to the failure area. For the cylindrical specimen with a prismatic section in the calibration area, two samples were separated so that they were containing failure surfaces of about 1 cm² with a volume of approximate 1 cm³.

![Figure 2](image2.png)

Figure 2. Samples debited and numbered for later identification.

For the investigation of uniaxial traction behaviour, the failure surfaces of three specimens with stress concentrators were analysed. The experiments were performed by scanning electronic microscopy using the SEM Quanta 200 3D Dual BEAMSEM / FIB electronic microscope at the following magnification powers: 50x, 1000x, and 5000x respectively. Additionally, a chemical analysis by the EDS-EDAX method was performed on the failure surfaces of the six samples in order to identify the mass percentages of the alloys.

3. Results and discussions
Using SEM analysis and EDS-EDAX method, the study of the failure surfaces was performed on a constant sample section for the following specimens 1.1, 1.2, 9.1, 9.2, 11.1 and 11.2. The results are portrayed in figures 3 to 9.

3.1. SEM Analysis

In the case of sample 1.1 (figure 3), the failure limits are relatively parallel. The surface presents failure macules that arise from the deformation process, some of which are highlighted as the brittle failure surface as the result of the avulsion phenomenon.

![Figure 3. Failure surface micrograph for the 1.1 specimen.](image)

The microscopic analysis of specimen 1.2 (figure 4) reveals the propagation lines of the material yielding, followed by failure. The rupture is influenced by the arrangement of the grain layers due to the lamination process of the plate from which the specimen is made. The presence of pores and inclusions is observed, which will influence the sliding of the layers, but also pieces of material left behind (debris) after failure. The dimensions of the inclusions are up to 2.84μm and the pores observed on the enlarged surface of 5000x have dimensions of 2.72μm, 3.50μm, 4.34μm and 5.78μm.

![Figure 4. Failure surface micrograph for the 1.2 specimen.](image)

In the conjugated surface of sample 1.1 (figure 4), the parallel strips are more clearly shown and it can be said that they were formed during rolling by developing sliding masses. The pulling phenomenon is characterized by the various inclusions in the material with the same chemical composition.

![Figure 5. Failure surface micrograph for the 9.1 specimen.](image)
As can be seen in figure 5, the micrograph of the specimen 9.1 shows a high degree of deformation with failure thresholds. The yielding has ductile characteristics such as the cone elements, as well as the existence of some constituents existing in the material. At higher magnification powers, a slip-free surface is observed without sliding layers. The latter are not highlighted because the surface area of the stress concentrator is smaller compared to the specimen 1, but also because of the brittle failure cause by the oxides (high O$_2$).

The same coupling elements are also observed on the sample surface 9.2 (figure 6) at the 1000x magnification power. The pores observed on the 5000x magnified surface are 10.26μm, 12.17μm and 18.90μm. The study of the pores was done in the area considered as representative and contained several voids that influenced the breaking process.

The failure mode of the 11.1 specimen (figure 7) is a brittle crystalline sludge free of slip slides because of the large amounts of SiO$_2$, MnO, Cu$_2$Al (extremely fragile compound), Mg$_2$O, so there are no macules slip or sliding strips. Although the aluminum alloy has predominantly ductile behavior, the brittle constituents influence it, so the failure has both ductile and brittle characteristics.

Specimen 11.2 shows AlFeO spherical particles and AlCu inclusions, whose dimensions are 2.59μm, 3.06μm, 3.34μm and 3.67μm. We can observe both the phenomenon of extraction, assigned to the brittle type of failure, and the cone-shaped formations that are the particular attributes of ductile failure.
The specimens analysed as a result of uniaxial traction failure developed triaxial stress states with ductile failure, although the fracture surfaces have a predominantly brittle appearance, with some areas and a ductile fracture. These discontinuities between the stress-strain characteristic diagram and the microscopic study are caused by the inhomogeneous structure of the material, which presents impurities and pores. The oxygen compound and the copper compound are brittle components that influenced the yielding and failure of the material.

3.2. EDS-EDAX
EDS-EDAX analysis was performed to determine the chemical composition of the samples. Each of the six failure surfaces was studied and the results were centralized in table 2. Comparing table 2 with table 1, where the chemical composition of the alloy was presented, consistent differences were observed and brittle compounds (that contain O$_2$) were identified in all of the samples.

Table 2. Weight percentage of the aluminium alloy.

| Sample | OK  | MgK | AlK  | CrK  | MnK | CuK | SiK | FeK |
|--------|-----|-----|------|------|-----|-----|-----|-----|
| 1.1    | 1.04| 1.02| 96.12| 0.42 | 1.4 | 0   | 0   | 0   |
| 1.2    | 1.25| 1.1 | 95.35| 0.34 | 1.5 | 0.47| 0   | 0   |
| 9.1    | 0.68| 2.03| 84.05| 0.28 | 1.27| 11.69| 0   | 0   |
| 9.2    | 0.72| 1.84| 84.07| 0.49 | 1.33| 11.55| 0   | 0   |
| 10.1   | 0.89| 2.92| 88.38| 7.14 | 0   | 0   | 0   | 0   |
| 10.2   | 1.22| 2.5 | 83.19| 0.97 | 7.14| 1.66| 3.32|

A sequential analysis was performed in order to determine the type of inclusions observed on the surface of the samples. Figure 9 depicts two EDS spectrums and both of them associate potassium amalgamates to the samples. The inclusions that were analysed sustain the fact that the failure took place not only cause of the loading conditions, but also due to the chemical composition of the material itself.

![Figure 9](image-url)

**Figure 9.** The EDS spectrum for an element of sample 1.2 and 11.2 respectively.

The work presented in this paper comes to complete previous papers that contained data from AEF analysis and the tensile tests of the specimens. The alloy should have a ductile type of failure, but as observed brittle elements were as well identified and they contributed into the yielding and then failure of the material.

4. Conclusions
The macroscopic and microscopic study by SEM analysis reveals the appearance of the fracture for the Al 96.5, Mg 3.1, Mn 0.25 and Cr 0.15 samples. Comparing the SEM images of the specimens, it is
clear that the Series 1 materials have brittle behaviour due to the presence of the sliding layers. Samples of the 9th and 11th series are shown to have a ductile behaviour, as evidenced by the cup-cone elements at the micrometric level. EDS-EDAX analysis has highlighted the chemical composition of the six surface areas with the presence of impurities, which are predominantly impurities of Al-Fe-O and Al-Cu. The impurity variation is between 2.59μm and 3.67μm, which contributes to breaking the material. In addition, the presence of copper in a significant amount influences the ductile failure (as seen in series 9 and series 11), whereas the presence of magnesium induces a brittle character in series 1.

5. References
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