Preparation and Examination of Aluminum Samples for Failure Analysis

By

Brad Peirson

School of Engineering Grand Valley State University

Laboratory Modules 1 & 2

EGR 250 – Materials Science and Engineering Section 1

Instructor: Dr. P.N. Anyalebechi

May 24, 2005

Abstract

A metallographic examination can be an essential tool in the examination of cause of product failure. The examination allows the key features of metallic structure to be observed and inferences to be made based upon that structure. In this laboratory a section a chair arm that had broken off was selected for examination. The specimen was cut, mounted and polished. After examination on a metallographic microscope the reason for the arm's failure was determined. The failure of the aluminum chair arm can be attributed to the size and frequency of pores, eutectic silicon particles and aluminumiron-silicon ([Al Fe Si]) constituent phase particles.

1.0 Introduction

In the hands of a skilled examiner, metallography is perhaps the most valuable tool available to an engineer [1]. Metallography is the study of the structure of metals. Knowledge of the structure can be invaluable in determining the reason of failure for a part or product. The area of a metallographic examination of greatest concern in terms of failure analysis is the microscopic examination. Microscopic examination is an invaluable tool that can be used to determine the class of material involved in the production, the method of manufacture, any heat treatment that may have been carried out as well as the characteristics of any cracks that may present [1].

The first stage of a microscopic examination is the selection of an appropriate specimen from the sample. The location of the specimen will be determined by the purpose of the examination. For instance, an examination of the effects of a corrosive material on a sample would require that the specimen be selected from an edge of the sample and not the interior [1]. The orientation of the specimen is also critical. In the case of a bar or wire a longitudinal section, a section that is parallel to the rolled surface, as well as a transverse section, one that is perpendicular to the rolled surface, must be selected [1]. This is caused by inherent differences in the structure of a rolled sample. Whatever the specimen, care must be taken to properly identify its location and orientation on either a diagram or picture of the sample [1].

Once a specimen is selected it must be cut from the sample. The two major categories of cutting methods are cold cutting and hot cutting. Cold cutting can mean a

1

variety of different saws, cutting wheels and other mechanical means. Whichever tool is used to achieve the desired cut there is a risk of damage to the structure of the metal. This risk is minimal if a sharp tool, a generous amount of lubrication and coolant or cutting fluid is used while cutting [1]. Hot cutting employs oxy/gas cutting torches. This method of cutting overheats the surface and alters the metal structure as far as 125mm from the edge [1]. For this reason, hot cutting is only used to take a small ingot from a bulk sample. This ingot can then be cut to size by different means [1].

After the specimen is cut from the sample it must be prepared for the examination. This usually involves two stages: mounting and polishing. The mounting operation is used to facilitate the handling of samples that would be difficult to handle during the polishing operation. Mounting can also be used if an automatic polisher is used that requires a standard sample size [1]. The two most common mounting techniques are mechanical mounts and thermoplastics [1]. Mechanical mounts are simply clamps that hold the specimen during polishing. When thermoplastics are employed they are heated sufficiently to melt them and the specimen is embedded in the plastic.

After the specimen is mounted through either means it must be polished. Polishing takes place over a series of bench grinders, hand grinders and polishing wheels. The sanding treatment gets progressively smaller at each stage and the specimen must be washed after each sanding to ensure than none of the particles from the last stage remain. The purpose of polishing the specimen is to achieve a scratch-free surface to be viewed under the metallographic microscope. Any scratches present will cause a distortion in the reflected light and the microscopic features will not be visible.

Once a specimen is polished it is examined on a metallographic microscope. This microscope differs from a biological microscope in that it detects light reflected from a specimen rather than light passing through it. The specimen is observed at the lowest magnification first to note the significant features. The power of the microscope can then be increased to reveal the structural details of the metal. This initial examination involves the as-polished specimen. Further observations can be made if the specimen is etched.

Etching is the process of immersing a specimen in a chemical etchant. Etchants are unique to the metal under examination. The proper etchant for a metal can be found

in reference materials. A chemical etchant attacks the boundaries between metal grains. This gives the boundaries a rougher appearance than the as-polished boundaries. This causes the boundaries to appear darker under the microscope. This helps to isolate the features of the metallic structure and make them easier to observe. The results of a microscopic examination are valuable only if the observations are correctly interpreted through knowledge of metallurgical properties [1].

Section 2.0 of this report outlines the procedure followed in the preparation and examination of the aluminum chair arm. Section 3.0 states the results of the microscopic examination of the chair arm specimen. Section 4.0 includes a discussion of the results and reasons for failure of the arm. Section 5.0 list the pertinent conclusions raised from the experiment. Section 6.0 is a works referenced section.

2.0 Experimental Procedure

2.1 Mounting and Polishing

A specimen was selected from an aluminum office chair arm that had failed. The section was a transverse section of the arm. It was first cut from the bulk sample using a cutting wheel. It was decided that the best face to mount was the transverse, h-shaped face. A schematic of the specimen with the mounted surface highlighted is shown in figure 1.



Figure 1: Schematic of the Chosen Specimen

The specimen was mounted using bakelite thermoplastic. The specimen as placed in a LECO PR-25 mounting press as shown in figure 2.



Figure 2: LECO PR-25 mounting press

The specimen was set on the ram and the ram was lowered. The bakelite disc was then set on the specimen. The top enclosure was sealed and the heater was turned on. The ram control arm was used to maintain a constant pressure for the duration of the built in timer. When the timer sounded after approximately 10 minutes the enclosure was opened and the mounted specimen removed. In order to speed up the cooling process the mounted specimen was quenched in the laboratory sink.

When the specimen had cooled it was taken to the bench grinder. The rough edges of the bakelite material were removed first to ensure that they wouldn't damage the paper on the hand grinders. The specimen itself was then ground to remove the rough scratches from cutting the specimen from the sample. The specimen was then taken to the hand grinders. Figure 3 shows a hand grinder similar to the one used to polish the specimen.



Figure 3: LECO DS-20 Grinder

The grinder was fitted with a series of silicon carbide (SiC) papers. The specimen was ground on the 240, 320, 400 and 600 sieve size papers for approximately five minutes apiece. All four papers were lubricated with a stream of water and the specimen was thoroughly washed between each grinding to prevent larger SiC grains being carried on to the next grinding. After the final hand grinding the specimen was washed and air dried before being taken to the polishing wheels.

The specimen was polished first on a wheel with 6µm diamond paste and then with 0.5µm diamond past. The specimen was polished for approximately 15 minutes per wheel. Both wheels were lubricated with white spirits during polishing. The specimen was washed with water and alcohol and dried with a hair dryer after each polishing wheel.

2.2 Etching and Examination

The specimen was placed on the metallurgical microscope for examination. The specimen was examined at 50x magnification in order to observe major features of the structure. The magnification was then increased to 500x in order to observe the finer details of the structure. Photomicrographs were taken at each magnification as documentation of the features observed at both stages.

The specimen was then taken to the fume hood for etching. The specimen was immersed in a 0.5% hydro-fluoric acid (HF) for a few seconds. The specimen was then removed from the etchant and immediately rinsed with water and alcohol. After the alcohol rinse the specimen was dried with the hair drier. The etched specimen was returned to the metallurgical microscope for further examination.

The specimen was examined first at the 50x magnification setting to observe the larger features of the structure. The magnification was again increased to 500x to observe the finer features. Photomicrographs were again taken at both stages as a record of the observations. Once the photomicrographs were taken, the reason for failure of the chair arms was determined.

3.0 Results

When polishing was completed, it was determined that the specimen had too many scratches to make any accurate observations. The specimen was re-polished at the 0.5µm polishing wheel for ten minutes in an attempt to remove the scratches. This attempt also failed. Finally groups C and D decided to combine and use group D's specimen for the observations.

The first photomicrograph of this specimen is shown in figure 4.



Figure 4: Photomicrograph of as-polished specimen at 50x magnification

Figure 4 shows that at the 50x setting, the structure of the as-polished specimen cannot be determined. The photomicrograph did reveal that the aluminum specimen was extremely porous.

Figure 5 shows the second photomicrograph of the specimen.



Figure 5: Photomicrograph of as-polished specimen at 500x magnification

At the 500x level the grains of the aluminum could be seen. The grain boundaries, however, were not clearly delineated. The porosity evidenced in figure 4 was also seen in this photomicrograph. This photomicrograph showed the [Al Fe Si] constituent phase particles. As they are not clearly delineated in figure 5 these particles have been circled.

The specimen was then etched in the 0.5% HF solution. After etching and drying the specimen was re-examined on the microscope. The first post etch photomicrograph is given in figure 6.



Figure 6: Etched photomicrograph at 50x magnification

After etching the cell boundaries were clearly delineated. This is clearly shown in figure 6. The clearly defined cell boundaries also allowed the relatively fine structure of the specimen to be observed. Figure 6 also shows the porosity of the specimen. The 500x post-etch photomicrograph is shown in figure 7.



Figure 7: Etched photomicrograph at 500x magnification

Figure 7 shows the clearly defined cell boundaries. It also contained evidence of the specimen's porosity. The [Al Fe Si] constituent phase particles were also more clearly defined after etching. Another structure visible in the post-etch photomicrograph were the needle-like eutectic silicon particles.

4.0 Discussion

4.1 Mounting and Polishing

From the failure of group C's attempt at polishing it was determined that extreme care should be taken at every phase of the polishing operation. It would be possible to improve the end results of the polishing operation if the groups would visually check for the absence of scratches at every step. Thorough washing was absolutely necessary between each step also. Another way to improve the final results of polishing would be to incorporate the metallographic microscope at various stages throughout the polishing operation. This would ensure that the major scratches from prior stages had all been removed. Over all it was determined that proper polishing technique requires patience and practice.

4.2 Etching and Examination

The appearance of slightly blurred cell boundaries suggests that the alloy had been heat treated. The etching process appeared to have been carried out correctly as the boundaries became clearly defined when removed from the etchant.

The examination showed several key features of the aluminum specimen. The relatively fine structure of the aluminum suggest a rapid cooling time. In this case the molten aluminum would not have had time to form larger cells. The examination also revealed that the specimen was extremely porous. This was likely caused by hydrogen gas dissolving in the liquid aluminum. As the metal began to freeze the hydrogen gas would have been expelled form the solid aluminum into the remaining liquid. This hydrogen would cause bubbles which translate to pores when the sample hardens completely.

The examination also revealed the formation of [Al Fe Si] constituent phase particles. The particles in this specimen were extremely coarse, where they should have been barely detectable between the cells. The presence of such coarse particles suggests that the molten metal was allowed to cool significantly before it was cast. This would allow the coarse particles to form before the metal was out into the mold.

A third feature observed in the examination was eutectic silicon particles. These particles appeared as needle like formations of roughly the same color as the constituent phase particles on the photomicrographs. The formation of these particles suggests that this aluminum alloy contains no strontium.

4.3 Failure Analysis

The reason for failure of this specimen was determined to be a combination of three of the features observed during the examination. The most obvious contributor seems to be the porosity. This specimen was more porous than most cast metals. This porosity translated to less cross-sectional area to distribute the force applied to the arm of the chair. The coarse [Al Fe Si] constituent phase particles as well as the coarse eutectic silicon particles also seem to have contributed to the failure. The large size and great frequency of both particles would have weakened the bonds between the metal's cells.

5.0 Conclusions

1. The polishing process must be carried out with patience. Accurate results seem to require both patience and practice of the techniques.

2. A specimen that has been poorly polished will be unrecognizable when placed on the metallographic microscope.

3. Etching does indeed make the cell boundaries easier to distinguish.

4. The presence of large, frequent pores and course particles likely led to failure of the chair arm.

5. Metallographic examination can reveal many different elements of a metallic structure that can be used as evidence of why a product failed.

6.0 References

 Anyalebechi, Dr. P.N. <u>Essentials of Materials Science & Engineering Laboratory</u> <u>Manual</u>. Grand Valley State University Padnos School of Engineering & Computing – School of Engineering, 2005, pp. 20-39.