Development and Characterization of Al-15\% Mo Binary Alloys

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Abstract. In this work, a binary Al-Mo alloy was made from aluminum and molybdenum elementary powder. The carefully homogenized samples were mechanically compacted to obtain a pastille of circular cross section ($\Phi = 15$ mm X 10 mm) under a load of 10 tons. Subsequently, the Al-Mo sample was melted in a high-frequency induction melting furnace. The resulting sample was polished and etched with a Keller reagent. The sample was subjected to metallographic observation on an optical and scanning electron microscope. An EDS analysis was performed on the different areas of the microstructure. A diffraction X-ray characterization was also performed in order to identify the different existing phases. This study showed that the studied alloy presents a microstructure with needle-like and plate-like precipitations of different sizes homogeneously distributed and randomly oriented. The EDS microanalysis showed the presence of the Al$5$Mo phase and an alpha aluminum matrix with a proportion of molybdenum. X-ray diffraction revealed the presence of several AL-Mo phases' namely alpha aluminum, molybdenum, Al$12$Mo, Al$8$Mo$3$ and Al$3$Mo.

1. Introduction
Numerous studies have been carried out on the Al-Mo system [1–4]. As this latter was insufficiently explored, the available data were not sufficient to identify the existing phases. This state could be gradually overcome due to the availability of different characterization techniques. This system was critically evaluated by Brewer and Schuster [5, 6]. These investigations have known notable advances thereafter where experimental studies were carried out by Schuster and Eumann on the aluminum rich region (Al-Mo$3$Al$8$) [7, 8]. Research on the aluminum-rich part was extended to higher molybdenum contents (33\% at. Mo) by Sperner in 1959 [9]. This work was subsequently detailed by Poetschke [10] for molybdenum contents ranging between 6 and 50\% by weight. In this study, the researchers used isothermal heat treatments and X-ray technique to correct the formula of MoAl$2$ to Mo$3$Al$8$. In 1962, Poetschke et al. did not lead to a rationalization of their conclusions on the form of the phase diagram studied. In this context,
Walford in 1964 and Brewer et al. in 1980 [5, 11], on the basis of the results provided by Poetschke, obtained different results. Using optical metallography, XRD and hardness measurement, Van Tendeloo in 1975 [12] and Belyaeva in 1967 [13] investigated about 20 alloys containing 0 to 33% in Mo using DTA for cooling the Debye process- Scherrer XRD, optical metallography. These authors maintained a “MoAl2” phase in their phase diagrams and therefore ignored the Poetschke data. In 1991, Schuster et al. examined 15 alloys containing 12 to 28% in Mo (made up of 99.8% by mass of pure Al and 99.9% by mass of pure Mo) using DTA for heating and Guinier XRD of alloys annealed. Ten intermetallic phases were observed. Recently in 2006 Eumann et al. [8] reexamined the Al-rich region (Al-Mo3Al8). A series of alloys containing 3.9 to 75.0% Mo was prepared by melting by crucible-free levitation following which most of the phases and phase equilibria were confirmed. A preliminary diagram of the partial system between 46 and 100 of molybdenum highlighting the MoAl3 phase, its behavior at peritectic fusion, and also the existence of a eutectic between MoAl3 and the next phase richer in Al was presented by Ham in 1950 and 1951 [14]. This scheme was approved by Sperner in 1959 [9] through observations on optical microscope of four alloys. In addition to the Mo3Al8 phase, the existence of the MoAl2 phase was reported by Belyaeva in 1967 [13]. MoAl2 coexists at 900 and 1000 °C with Mo3Al8 on the Al rich side and at 1100°C with MoAl3 on the Mo rich side. Two intermediate high-temperature phases, Mo63Al37 and MoAl were reported by Rexer in 1971 [15] during investigations carried out on numerous balanced alloys containing 25 to 100% molybdenum produced from aluminum powders and 99.99% pure molybdenum. These two phases subsequently decomposed at 1490°C and 1470°C respectively. In this work, Rexer claimed that these two phases melted congruently. This work did not investigate the areas around 50% molybdenum but it cannot exclude the formation of a MoAl peritectic phase.

The MoAl5 phase is a polymorphic phase of the Al5W type which is involved in two to three modifications [7, 10, 16]. Recent experimental work has not observed the polymorph MoAl5 (h2) at high temperature but has confirmed its presence at medium temperature MoAl5 (h1) as a metastable phase. The thermodynamic evaluations of the Al-Mo system was the subject of several research works [1, 2, 17-20]. Due to a lack of data on the stability and exact sequence of reactions Saunders did not consider the Mo4Al17, Mo5Al22, and MoAl3 phases when studying the Al-Mo system. This work deals with the elaboration and characterization of an aluminum alloy containing 15% molybdenum. The melting took place in a high-frequency induction furnace. Specimens were prepared from elemental aluminum and molybdenum powders that were compacted into tablet form.

2. Experimental techniques

The raw materials used for the preparation of the Al-15%Mo binary mixture include aluminum and molybdenum elementary powder. The powder was weighed on a precision microbalance of 10-3g in order to obtain the desired nominal composition. The base materials are homogenized and then compacted using a compacting press to obtain pastilles of circular cross section (Φ= 15 mm X 10 mm) under a load of 10 tons.

Figure 1: Compacting press and Al-Mo alloy
The fusion takes place in a high frequency induction furnace of the Lin Therm HG6000 type having a power of the order of 6KW (Fig. 2).

![Fig. 2: Set of sample, crucible and quartz tube placed inside the inductor](image)

The results presented in this work are obtained from raw samples. When melted, the cooled samples were prepared according to the conventional method. A Keller reagent was used as the etching solution in order to allow a good microstructure revelation as well as observation of the precipitation shape, orientation and size. The observations of the microstructure were carried out on an optical microscope of the Nikon Eclipse LV150N type and on a scanning electron microscope of the QUANTA 250 type.

The Vickers microhardness tests were carried out on a Matzuzawa SEIKI MXT70 microdurometer. The X-ray diffraction analysis was performed on a Brucker D8 advance type diffractometer of geometry (θ - 2θ) called Bragg-Brentano over an angular range of 0 - 120° in 2θ with a step of 0.02°. The radiation used is that of copper, the wavelength $\lambda_{cuk\alpha} = 1.54056$ A°. The identification of phases was performed by using ASTM files.

3. Results

3.1. Optical and SEM microscopy

Figure 3 (A and B) shows the optical microstructure of the binary Al-15%Mo alloy. The micrographs indicate clearly the shape and distribution of the precipitation. It illustrates the different precipitations which are distinguished by their shapes. Compacted aluminum and molybdenum powders melt and interact at high temperature to form a structure of an alpha-aluminum matrix on which AlxMoy inter-metallics are precipitated. These precipitations have a needle and platelet shape, are randomly oriented and homogeneously distributed in the matrix.
Figure 3: Optical micrograph of the sample (AL-Mo).
EDS analysis (figure 4) performed on different areas of the microstructure show that molybdenum is distributed between the matrix and the precipitation. During melting, small proportions of molybdenum are dissolved in aluminum. The remaining molybdenum interacted with the aluminum to form different precipitations. The precipitate appears on the microstructure with three levels of light degraded colours. This can be explained by the presence of molybdenum in different grades. The results of the EDS analyses carried out on the precipitates (Fig. A, B, C, D, E and F) show that the molybdenum content is concentrated in the clearest precipitates.

3.2. Microhardness
The results of the hardness test carried out on the studied alloy are presented in Table 1. A clear difference between the microhardness values measured on the matrix and on the precipitates is noted. This is explained by the high concentration of molybdenum in the precipitates. The matrix, which is made of aluminum, can only contain molybdenum at very limited contents. The measured values range from 48 to 73 for the matrix and from 218 to 299 for the precipitates. This difference in microhardness is attributed to the different molybdenum concentration in the matrix and in the precipitates.

| Elements | % At. |
|----------|-------|
| Al-K     | 80.01 |
| Mo-K     | 2.25  |
| O-K      | 17.74 |

Table 1: Microhardness values

| (Al-MB) | HV0.5 Matrix | HV0.5 Precipitates |
|---------|--------------|--------------------|
| 59.9    | 252.5        |                    |
| 68.2    | 231.5        |                    |
| 70.3    | 287.9        |                    |
| 50.6    | 218.6        |                    |
| 73.9    | 223.5        |                    |
| 60.6    | 292          |                    |
| 59.2    | 299          |                    |
| 66.0    | 268          |                    |
| 48.0    | 268.5        |                    |
| 64      | 256.7        |                    |
3.3. X-ray diffraction

The X-ray diffraction pattern of the raw sample is shown in Figure 6. The identification of the phases was carried out using the ASTM files. The X-ray diffraction technique showed the presence of numerous phases other than Al5Mo. The revealed phases are: alpha aluminum, molybdenum, Al12Mo, Al8Mo3 and Al3Mo. According to J.C. Schuster and al. [3], Al5Mo exists in three forms, belonging to different space groups and arising at three different temperature levels: (i) The high-temperature form; (ii) the low-temperature form and (iii) the intermediate temperature form. It has a hexagonal structure.

![XRD pattern of the Al-15%Mo alloy](image)

**Fig. 5:** XRD pattern of the Al-15%Mo alloy

4. Conclusion

- An Al-15%Mo alloy consisting of elementary aluminum and molybdenum powder was produced in a high-frequency induction furnace.
- The Al-Mo system has been investigated by several research studies. It has been updated with new results, some of which have confirmed the presence of certain phases and others have practically provided new knowledge and details about the system.
- The metallographic analysis showed the presence of precipitation of intermetallic phases of different morphologies and dimensions randomly oriented and homogeneously distributed on the microstructure.
- Micro-analyses performed by EDS on precipitation show slight differences in molybdenum values which may confirm the existence of the Al5Mo phase.
- The microhardness values measured on the precipitates are higher than those measured on the matrix.
- X-ray diffraction showed the presence of the following phases: alpha aluminum, molybdenum, Al12Mo, Al8Mo3, Al5Mo and Al3Mo.
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