T.E.M study of the decomposition of some rf-sputtered Aluminium-Iron thin films.

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Abstract The complexity of the equilibrium phase diagram of aluminium-iron, in particular on the aluminium rich side, makes difficult any prevision of the decomposition behavior of the sputtered thin films deposits. For some of them, the decomposition is marked by the appearance of new intermetallic compounds resulting from the crystallization of an amorphous or nanocrystalline metastable phase.

1.Introduction

Thin films of alloys and intermetallic compounds have a wide range of applications. The role of these compounds can have a particular interest in the improvement of the intrinsic properties of material by conferring to it a better thermal stability, a good corrosion resistance and to hot oxidation. These include conducting barrier layers and passivation layers in microelectronic devices. The intermetallic compounds NiAl [1] and FeAl are under active investigation as structural materials for high-temperature applications Moreover, the equilibrium phase diagram of aluminium-iron reveals that aluminium rich phases such as Al_2Fe, Al_5Fe_2 and Al_3Fe could be competitive with nickel or titanium aluminides phases considered for their good oxidation resistance [2]. We study the decomposition behavior and phase transformation of two sputter deposited aluminium-iron thin films containing respectively 18,5 and 47,5at%Fe, after annealing at 500°C .

2.Experimental procedure

The thin films were elaborated within an installation whose cathode is polarized in radio frequency .The vacuum chamber has a volume of approximately 40 liters. The pumping installation consists of a vane pump and a secondary turbo molecular pump making it possible to reach a limiting vacuum of 10^{-4} Pa. horizontal cathode magnetron, 51 mms in diameter, consists of two concentric magnets of opposite polarity, the target ensuring the sealing of the device is cooled by water circulation

The thin films which we elaborated were characterized by x-ray diffraction (XRD), transmission electron microscopy (T.E.M) and microanalysis with X-EDS.

3.Results and discussion

Let us recall that at the as-deposited state Al-18,5at%Fe thin film is single-phase amorphous and present a pre-peak of diffusion (Figure 1a) characteristic of a strong interaction between iron and aluminium atoms. The crystallization of this thin film followed by diffraction of x-rays and transmission electronic microscopy leads to equilibrium Al_3Fe phase (Figure 1b) and compared with those calculated from the crystallographic data of Al_3Fe phase[3].

The electron micro diffraction, by the analysis of the only pattern of zone axis [104] (Figure 1c), allowed us to determine without ambiguity the crystalline system (monoclinic),the class of Laue (2/m) and the Bravais mode (C) of Al_3Fe phase.

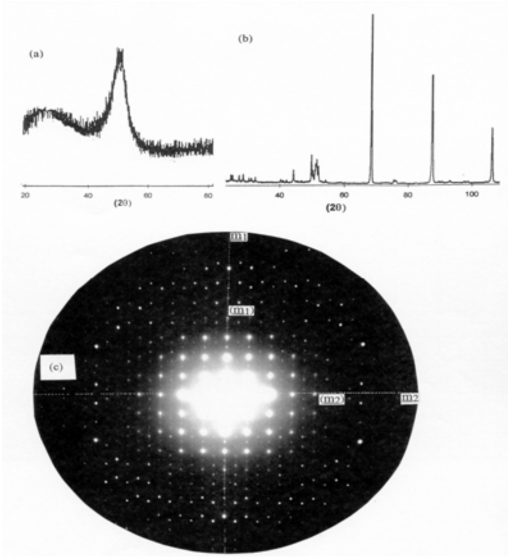

Fig.1. Al-18,5at%Fe deposit
(a):(as-sputtered)x-ray diffraction patter n(b):(Annealed 1h –500°C) x-ray diffraction pattern (c):(Annealed 1h –500°C) electron diffraction pattern, zone axis [104].

The decomposition of Al-47,5at%Fe deposit initially ordered in the form of a B_2 CsCl type structure, leads to a structural evolution which is characterized by a recrystallization with polyhedric arrangement of the grains (Figure 2a).The x-ray diffraction (Figure 2c) permit to measure with precision the interplanar spacing of various peaks which appear. We note that except the fundamental and superstructure lines belonging to the ordered B_2 (FeAl), several others are observable (16°32, 19°65, 21°74, 32°99). The electron diffraction (Figure 2b) lets appear an additional ring, among others, to which one can allot an interplanar spacing of 0,257 nm. Already observed in the as-deposited state of pulverisation[4], this ring was associated to the (0001) plan of the athermal omega phase, here it corresponds to the(0003) plan of a rhomboedral phase because several other distances characteristic of this phase were observed. $a_R \approx 0{,}89$ nm et $\alpha \approx 111° 8'$ for rhomboedral cell. or $a_H \approx 1{,}47$nm et $c_H \approx 0{,}79$nm hexagonal cell. [5].

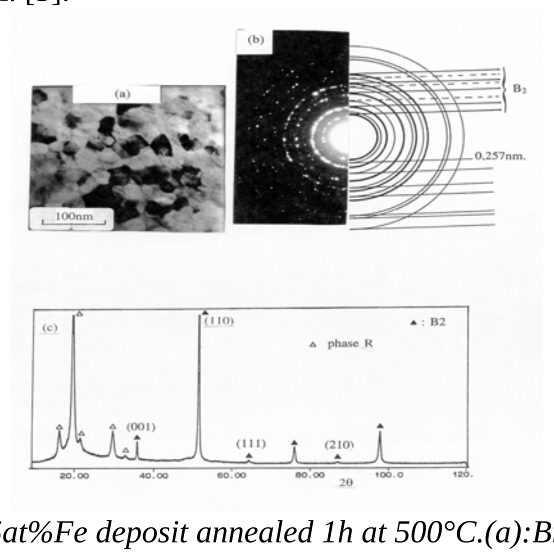

Fig.2. Al-47,5at%Fe deposit annealed 1h at 500°C.(a):Bright field electron micrograph (b):Associated SAED pattern(c): X-ray diffraction pattern (B_2 phase and R phase).

Indeed six interreticular spacing carried in table(4) would belong to rhomboedral phase (R) Consequently, Al-47,5at%Fe deposit would consist of a mixture of B_2 and rhomboedral (R)phase. The question of a possible link between the presence of omega phase detected in the as-deposited state[4] and the formation of the rhomboedral phase after an annealing at 500°C arises.

4.Conclusion

The decomposition after annealing at 500°C(1h), of metastable aluminium-iron thin films containing 18,5 and 47,5at%Fe was studied by means of transmission electron microscopy and x-ray diffraction .The principal results are as follows:

Amorphous Al-18,5%atFe thin films initially single-phase decomposes after crystallization to give rise to stable intermetallic compound Al_3Fe.

Al-47,5%Fe thin film, initially ordered in the form of B_2 CsCl type with traces of omega phase, leads to the formation of the rhomboedral phase undoubtedly initiated by transformation of the omega phase with which it presents a structural similarity.

5.References

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