OPTICAL MICROSCOPY IN THE STUDY OF METALLIC ALLOYS

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ABSTRACT
The optical microscopy is a traditional, very important tool for the study of materials, in particular of metallic alloys. Even though electron microscopy techniques have evolved to forms increasingly more sophisticated, optical microscopy remains as a tool indicated for the study of a broad spectrum of topics linked to the physical metallurgy, such as solidification, phase transformations products, grain structures, fracture, among others. The most important application of light microscopy is the determination of the structural phases present and the constitution of the bulk of the metal. These observations are of practical importance because the structure and constitution have a strong influence on the behavior of the material.
In this work, we present numerous examples of optical microscopy applied to the study of metallic alloys. In particular, the phase transformations taking place in Cu-based shape memory alloys, and the analysis of structures formed during directional solidification of dilute alloys. In all the cases, the specimens were carefully prepared for optical examination in a Reichert MeF2 Optical Microscope, with a photographic system put on. The studied alloys and the metallographic procedures are mentioned opportunely.

Keywords: optical microscopy, metallic alloys, phase transformations, directional solidification.

MICROSCOPIA ÓPTICA APLICADA AL ESTUDIO DE ALEACIONES METÁLICAS

RESUMEN
La microscopia óptica es una herramienta tradicional, muy importante, para el estudio de materiales, en particular, de aleaciones metálicas. Aún cuando técnicas de microscopia electrónica hayan evolucionado a formas cada vez más sofisticadas, la microscopia óptica sigue siendo la herramienta indicada para el estudio de un amplio espectro de temas vinculados con la metalurgia física, tales como estructuras de solidificación, productos de transformaciones de fase, estructuras de grano, superficies de fractura, entre otros. La más importante aplicación de la microscopía óptica es la determinación de las fases presentes y la constitución de la matriz metálica. Estas observaciones son de gran importancia práctica debido a su influencia en el comportamiento del material.
En este trabajo se presentan numerosos ejemplos de aplicación de la micrografía óptica en el estudio de aleaciones metálicas. En particular se abordan las transformaciones de fase que tienen lugar en aleaciones con memoria de forma base Cu y el análisis de las estructuras de solidificación unidireccional. En todos los casos, las muestras fueron cuidadosamente preparadas para ser observadas en un microscopio óptico Reichert MeF2, que cuenta con sistema adquisidor de imágenes. Las aleaciones estudiadas y los procedimientos metalográficos se mencionan oportunamente.

Palabras clave: microscopía óptica, aleaciones metálicas, transformaciones de fase, solidificación unidireccional.

INTRODUCTION

Although numerous sophisticated electron microscopy tools are now available, the light microscope remains as a very important technique for the study of a wide field in materials science. TEM and SEM overcome two of the most important limitations of the light microscope, resolution and depth of field; indeed the limitations of the TEM and SEM are the strong point of the light microscope and vice versa, so they are complementary rather than competitive. Optical microscopy is an efficient and inexpensive means for examining features of a material over a wide range of magnifications. The optical microscope reveals features to the trained observer that cannot be determined from a scanning electron microscope (SEM) examination; for example, color, relative opacity, birefringence and refractive index.

The optical microscope is an important tool of the metallurgist from both the scientific and technical standpoints. It is used to determine grain size and the size, shape, and distribution of various phases and inclusions which have a great effect on the mechanical properties of metals. The microstructure will also reveal the mechanical and thermal treatment of the metal. Optical investigations provide valuable microstructural information and can be used to select features requiring more detailed analysis. In many instances the optical investigation is sufficient to resolve the specific problem under investigation thus eliminating the need for more costly methods of analysis. Furthermore, microscopic studies depend upon the care taken in sample preparation; they must be properly prepared to reveal, through selective attacks or colored phase process, specific details of the studied structure. The most expensive microscope will not reveal the structure of a sample that has been poorly prepared. Afterwards, the surface is observed in response to the different illumination conditions (bright field, dark field, polarized light, interference contrast) which highlight specific features or structures of a material. Nowadays, the implementation of modern images scanning and processing techniques facilitates and improves the collection of information.

In this work, we present examples of light microscopy applications in the study of phase transformation in Cu based shape memory alloys and in the structures analysis during directional solidification of dilute alloys.

MATERIALS AND METHODS

In all the cases, the specimens were carefully prepared for optical examination in a Reichert MeF2 Optical Microscope, with a photographic system put on. The studied alloys and the metallographic procedures are mentioned below.

RESULTS AND DISCUSSION

PHASE TRANSFORMATION IN CU-BASED SM ALLOYS

The Cu-based alloys have found widespread technological applications owing to their shape memory effect and pseudoelasticity. They are particularly interesting because of their low cost and relative easiness of processing. However, they show considerable aging effects that have constrained their potential applications.

The parent β phase of the shape memory alloys (SMA) while stable at high temperatures is not often thermodynamically stable at lower temperatures; it can undergo diffusion decomposition to the equilibrium phases, generally α and γ, during cooling or by isothermal treatments at moderate temperatures [1,2]. This fact leads to a degradation of the shape memory capacity and a change in martensitic transformation temperatures [3-5].

While electronic microscopy is important in the first stages of decomposition, as the γ precipitates increase their size and amount, the optical microscopy is revealed as the adequate observation tool (Figure 1a). The morphology evolution of the precipitation phases could be deeply analyzed, as well as the process kinetics [6]. The same is true for CuAlBe alloys [7] and others CuAl-based ones.

The γ phase is just well revealed with a polishing procedure using an electrolytic solution of chromium trioxide saturated in phosphoric acid [8]. This electrolyte is sometimes used, with lower voltages, for electropolishing CuZnAl alloys for TEM observations. However it is not generally effective for revealing other phases, like α; then it is necessary to perform a complementary procedure. Immersing the specimen in a chemical reagent constituted by a mixture of ferric chloride and hydrochloric acid dissolved in ethyl alcohol, shows to be very helpful for this purpose (Figure 1b). The morphology of the α phase depends on many factors; one of them is the aging temperature. The plate shaped morphology, sometimes referred to bainite**,** was observed during isothermal aging at low temperatures (Figure 2a), while at higher temperatures a rod-like phase was formed (Figure 2b).

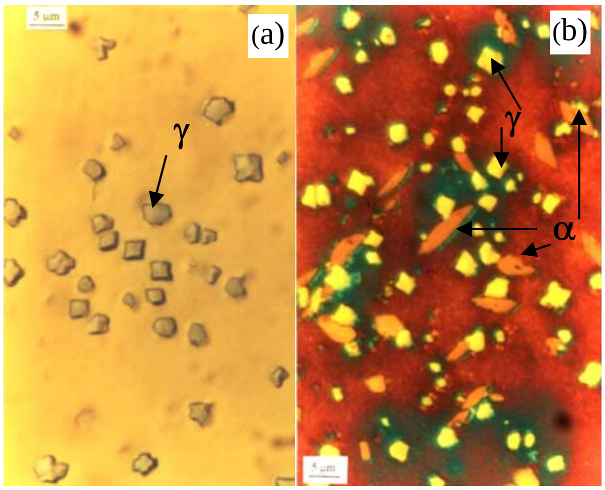

Fig. 1. (a) γ precipitates in CuZnAl alloy; (b) the same chemically etched and colored, improving α phase observation. (note: γ in yellow; α in orange)

In order to improve the detection of the phases, achieve a higher accuracy in the identification, and mainly, in the quantitative image analysis, the effectiveness of the color metallography was proved (Figure 1b - 2). This procedure is a color etching one, using an aqueous reagent containing sodium thiosulfate, lead acetate and citric acid [**¡Error! Marcador no definido.**].

During cooling from high temperature, the β phase undergoes order transitions; in particular in CuAl-based alloys, an ordering reaction from a disordered A2 (bcc) structure to an ordered DO_3 one occurs near 800 K. By optical microscopy, rosette-shaped regions between the γ_2 precipitates, and envelopes around them, could be observed (Figure 3). These regions constitute domains of β_1, that is DO_3 ordered β-phase [**¡Error! Marcador no definido.**].

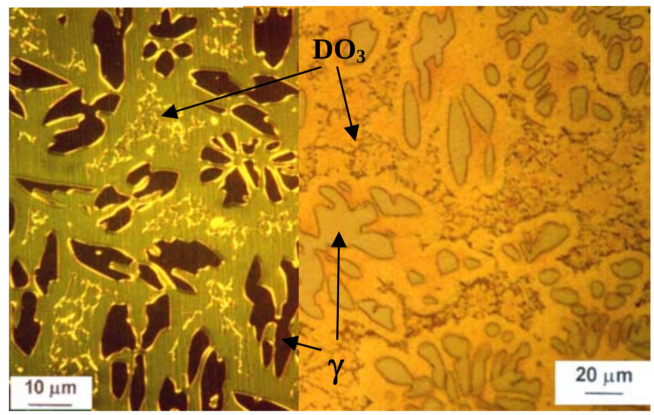

Fig. 3. DO_3 order domains in CuAlBe alloy; polarized light enhances the detection.

The characteristic lens shaped martensitic microstructure is formed as the result of a diffusionless phase transformation which involves large scale lattice variant and invariant shearing. The heterogeneous domain microstructure which results can be visible by etching, the

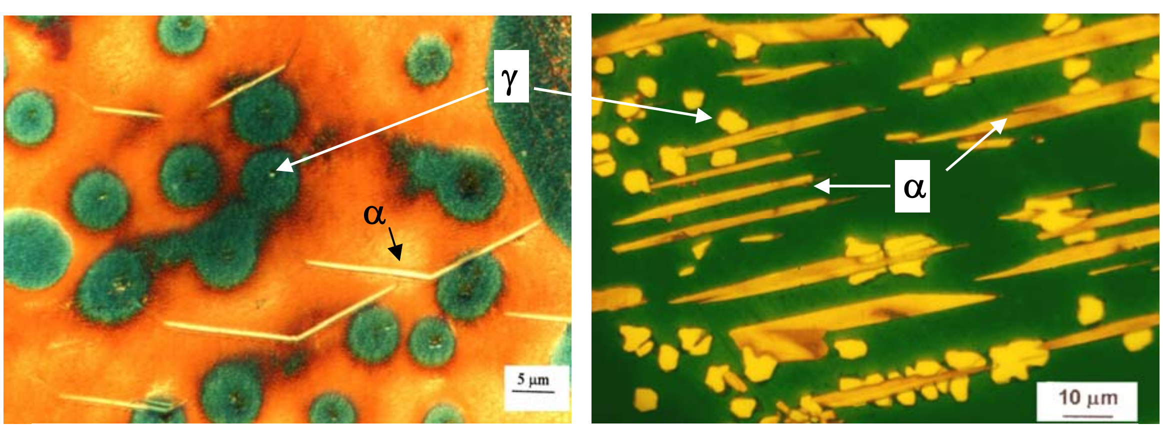

Fig. 2. Color metallographies of α phase formed in CuZnAl alloys during isothermal treatments.

previously mentioned electrolytic solution of chromium trioxide saturated in phosphoric acid could be used, and light microscopy is a good technique to study them (Figure 4). Polarized light and contrast effects are of great uselessness in this case.

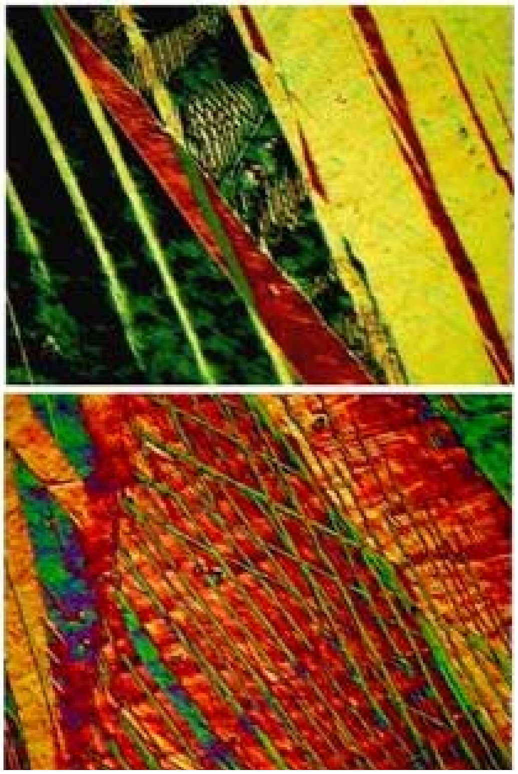

Fig. 4. Martensite plates in a CuAlMn alloy.

MICROSTRUCTURES FORMATION DURING DIRECTIONAL SOLIDIFICATION

Optical microscopy is a fundamental tool to the study of solidification, where the structures have a typical size of hundred of microns, and details are of tenths. Electrolytic polishing, etching and oxidation are a useful complement to the first interpretation of microsegregation and pattern formation.

It is well known that segregation plays a fundamental role in the selection of the microstructures during directional solidification of dilute alloys [9]. Particularly, solute increases the composition of liquid in front of the interface, with a characteristically pattern which depends on other solidification parameters. During the cellular stage of growth, center of cells or broad dendrites growth with a composition lesser than nominal composition C_0 by a rate given by partition coefficient k_0, that means

$$C_t = k_0 C_0$$

where C_t is the composition at cell tip. Typical values for k_0 are in the order of 0,1 – 0,2 [10]. By other side, nominal composition is chosen depending on the system under study, but could be fall in two important categories for binary alloys: composition bellow or above the maximum solubility range, which means the possible formation of a second phase under equilibrium considerations [11]. In the former case, the excess of solute is built up in front of the interface and located in the cellular walls [9], with a composition that can reach a value of

$$C_t = k_0 / C_0$$

that is one or more magnitude order above C_t in the same phase. In the Figure 5 can be see a cellular wall which presents a regular lateral perturbation as a consequence of the lateral growth. The specimen is Al-0,2 wt. %Cu directionally grown and the figure shows a transversal cut of the sample. The preparation was achieved through mechanical and electrolytic polish, with a 2-butoxi-ethanol (80%), glycerol (10%) and percloric acid (10%) electrolyte, at 33-40VCC with purity aluminum anode. A posterior chemical etching with dilute Keller solution was used to improve the contrast.

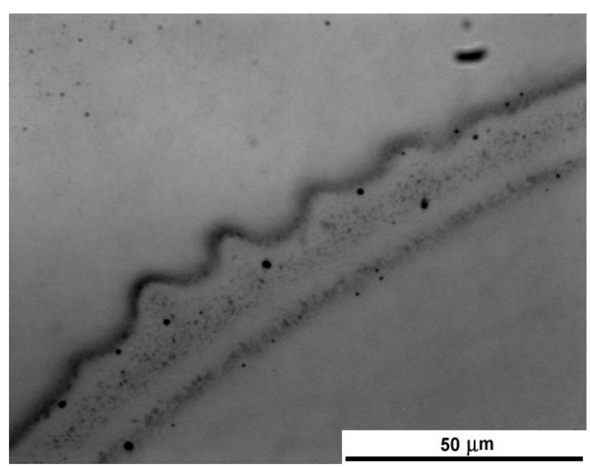

Fig. 5. Al-0,2 %Cu, view of cellular wall, without the presence of a second phase.

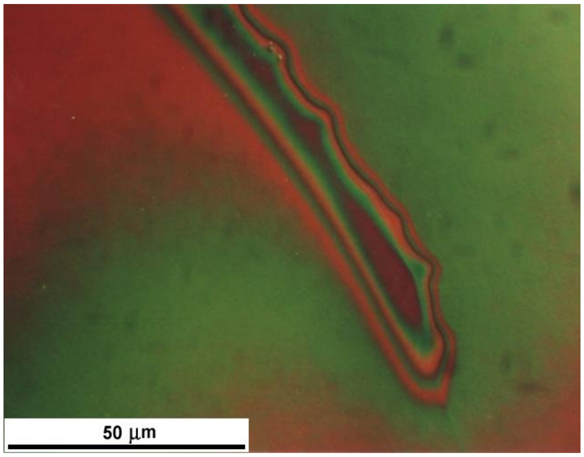

Fig. 6. Anodic Oxidation in Al-0,2%Cu, showing a composition profile inside of the cellular wall.

Figure 6, shows a similar microstructure, but in this case, the sample is oxidized by a water (98%) and sulphuric acid electrolyte (2%). Anodizing conditions are very sensitive, being needed several attempts to obtain successfully images. This kind of anodizing brings us a qualitative segregation map when is observed with polarized light.

For samples with composition close or above the maximum solubility limit, often is observed that a second phase appear, even for concentration below eutectic composition. In these cases the composition of cellular walls or vertex nodes can reach the eutectic compositions [**¡Error! Marcador no definido.**]. Figure 7 shows this situation.

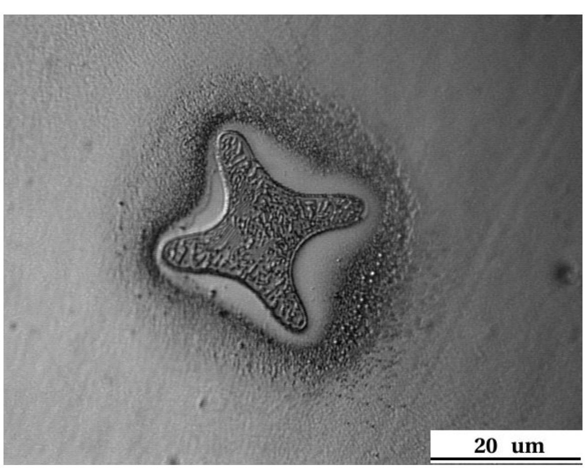

Fig. 7. Al-1%Cu, view of a cellular vertex far from the S-L interface.

The sample is prepared as for Figure 5, but the chemical etching used is even dilute in three parts of distilled water to preserve the oxidation of eutectic node.

In the micrograph can be seen that surrounding the eutectic there exist a white zone, which is formed by back-diffusion during the solidification of the node

In systems where second phase does not shows a precipitation, like Sn-Pb, the cellular walls shows a different structure. The Figure 8 shows a micrograph obtained by pasive oxidation of electropolished surface. The oxidation was achieved by oxygenate water (30 vol.) during few seconds.

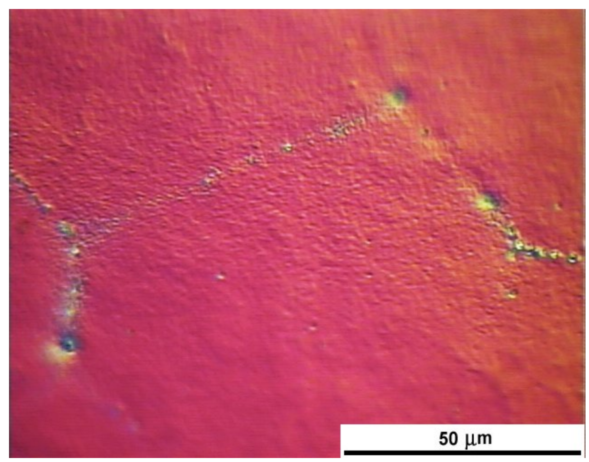

Fig. 8. Sn-0,05%Pb, oxidized, the composition difference suggest the existence of a cellular wall.

CONCLUSIONS

Numerous examples of optical microscopy applied to the study of metallic alloys have been presented; in particular, those related with the phase transformations taking place in Cu-based shape memory alloys and the analysis of structures formed during directional solidification of dilute alloys. Optical microscopy resulted a valuable tool for materials science researcher in several interest fields. The examples used in the work showed several of them. Despite the importance that new techniques based in electron microscopy, the optical microscopy with the addition of acquired black and white or color images, is a very important technique that provides valuable microstructural information for the investigations.

ACKNOWLEDGEMENTS

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