Metallography of magnesium die-cast alloys

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Magnesium and its alloys, regardless of the processing procedures employed, are among the most difficult metallic specimens to prepare for microstructural examination. Mg and its alloys are low in hardness and strength but do contain precipitates that are much higher in hardness. This makes it difficult to eliminate scratches and matrix deformation and to control relief, that is, excessive height differences between the matrix and precipitates. Mg is also quite reactive and there is considerable debate over whether or not water must be eliminated from the final preparation step or steps. Although its crystal structure is hexagonal close-packed, Mg does not respond well to polarized light after polishing. The paper presents a new procedure for preparing Mg and its alloys that yields flat surfaces with no relief problems and minimal scratches and matrix deformation. Some comments about etchants are also made.

Parole chiave: magnesio e leghe, metallografia, pressocolata

INTRODUCTION

Preparation of magnesium and its alloys is rather difficult due to the low matrix hardness and the higher hardness of precipitate phases that lead to relief problems, and from the reactivity of the metal. Mechanical twinning may result during cutting, grinding, or handling if pressures are excessive. Final polishing and cleaning operations should avoid or minimize the use of water and a variety of solutions have been proposed. Pure magnesium is attacked more slowly by water while Mg alloys may exhibit much higher attack rates. Some authors state that water should not be used in any step and they use a 1-to-3 mixture of glycerol-to-ethanol or kerosene as the coolant even in the grinding steps. It is always best to grind with a coolant, as fine Mg dust is a fire hazard. Because of the presence of hard intermetallic phases, relief may be difficult to control, especially if napped cloths are used.

Cutting can introduce considerable damage to specimens and this can be a critical factor in obtaining properly polished specimens. Always use the cutting technique that produces the least amount of damage. An abrasive cut-off saw produces excellent results. Again, a coolant must be used when cutting to minimize damage produced from the heat generated during sectioning. Although magnesium and its alloys have a relatively low melting point, and the solution annealing and aging temperatures are lower, hot compression mounting can be used. However, if the specimen were in the as-solution annealed condition, without aging, it would be prudent to use a castable resin, such as epoxy, that generates very little heat during polymerization. The pressure used in hot compression mounting may induce mechanical twinning in high-purity magnesium.

A number of procedures have been published for mechanical polishing of Mg and Mg alloys. Traditional approaches utilize SiC paper to wet grind the specimens using a series of graded abrasive papers from about 240 to 600 grit (P280 to P1200), or even finer. Some metallographers have coated the SiC surfaces with wax to minimize embedment of abrasives. This was tried, and compared to non-coated SiC grinding. Embedding of SiC abrasive particles was not observed. It does not seem to be necessary to coat the SiC paper with wax. Water can be used to flush the grinding debris from the SiC paper surface and minimize heating of the specimen. This is followed by several stages of rough and fine polishing using two or more sizes of diamond abrasive. A coolant/lubricant must be used and these can be water-soluble or alcohol based.

Final polishing has always been a weak point in the process. Magnesium oxide was used but it is difficult to get good quality MgO and the particle size is 1 μm, which is too coarse. MgO is also hard on polishing cloths. MgO can be suspended in water, but it is best to avoid water. Alumina has been used as a final polishing abrasive, and it is available in sizes down to about 0.05 μm. It can be purchased as a powder, or as pre-mixed slurries but these are aqueous. Alumina has been used as an aqueous suspension, with additions of soap, and has been mixed with alcohol or ethylene glycol. I have used colloidal silica and this has been effective with pure magnesium but it etches magnesium alloys. Cleaning can be done with water between grinding steps, and after diamond polishing, but generally water is avoided after the final polishing step. Instead, alcohol or other solvents are employed.

PREPARATION EXPERIMENTS

A number of experiments were performed in our laboratory to develop a practical mechanical polishing procedure for Mg and its alloys. A wide variety of specimens, cast and wrought, were tried starting with pure Mg and then following with popular alloys. First, we tried using one or two SiC steps and then going through a series of diamond (synthetic polycrystalline) polishing steps using Metadi® Supreme aqueous suspensions at 9-, 3- and 1-μm mean particle sizes. The cloths used were napless, flat pads and several were evaluated. The synthetic chemotextile Texmet® 1000 pad was found to give excellent results. Napped cloths should be avoided as they will produce excessive relief and may lead to other problems, such as drag or pull out. While the aqueous diamond suspensions yielded good results, oil-based suspensions (synthetic monocrystalline diamond) were tried to see if avoiding water would produce better results. In general, no evidence was obtained to suggest that the wa-

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Surface	Abrasive/Size	Load Lb. (N)	Speed rpm/Direction	Time (minutes)
Carbimet waterproof paper	320-grit SiC (P400) water cooled	5 (22)	250 Comp.	Until Plane
Texmet 1000 pad	9-µm Metadi oil-based diamond slurry	5(22)	150 Contra	6
Texmet 1000 pad	3-µm Metadi oil-based diamond slurry	5 (22)	120 Contra	5
Texmet 1000 pad	1-µm Metadi oil-based diamond slurry	5 (22)	120 Contra	4
Chemomet cloth	0.05-µm Masterpolish or Masterprep abrasives	3 (13)	120 Contra	1.5-3

Note: In contra rotation the platen and the specimen holder rotate in opposite directions while in comp. (complementary) rotation, they rotate in the same direction.

Table 1. Five-Step Mechanical Polishing Procedure for Magnesium Alloys.

Tabella I - Procedimento per la lucidatura di leghe di magnesio a 5 passaggi

Name	Composition	Comments
Glycol	1 mL HNO_3 24 mL Water 75 mL Ethylene Glycol 20 mL Acetic Acid	General-purpose etch. Immerse for 3-5 seconds; rinse in water and dry.
Acetic Glycol	1 mL HNO_3 60 mL Ethylene Glycol 20 mL Water	General-purpose etch. Immerse for 1-3 seconds for cast alloys and up to 10 seconds for solution annealed alloys; rinse and dry.
Acetic-Picral	5 mL Acetic Acid 6 g Picric Acid 10 mL Water 100 mL Ethanol	Immerse until a brown film forms on the surface; rinse and dry. May reveal grain boundaries, mechanical twins and residual cold work.
Phospho-Picral	0.7 mL H3PO4 4-6 g Picric Acid 100 mL Ethanol	Immerse for 10-20 seconds, rinse and dry. Stains the matrix; massive $Mg_{17}Al_{12}$ phase remains white.
Hydrofluoric Acid	10 mL HF 90 mL Water	Immerse specimen for 1-2 seconds. Darkens $Mg_{17}Al_{12}$.

Table 2. Chemical Etchants Used in the Study.

Tabella 2 - Agenti d'attacco chimico utilizzati in questo studio

ter in the diamond suspensions attacked the microstructural constituents. However, the greater lubricity of the oil suspension appeared to yield a better surface finish with fewer, shallower scratches.

For final polishing, two cloths were tried. A medium-nap synthetic-suede Microcloth® pad and a napless, synthetic polyurethane Chemomet® pad were evaluated. It was thought that scratch control might be better with the softer Microcloth pad, while surface flatness (relief control) would be better with the Chemomet pad. However, no relief problems were encountered with either pad, but the results with the Chemomet pad were slightly better. Because the colloidal silica polishing suspension etched the Mg alloys, other suspensions were tried. Alumina is normally produced by the calcination process, but these abrasives always contain some degree of agglomeration. Recently, alumina made by the sol gel process has been developed, although only in very fine sizes. Masterprep™ alumina suspension is a sol-gel product with an average particle size of 0.05 µm. This abrasive produced satisfactory results. Another proprietary polishing suspension, Masterpolish® abrasive, was also tried. It is a viscous mixture of alumina and silica and is suspended in a mixture of water, petroleum distillates and propylene glycol. This abrasive also produced satisfactory results. For thin-walled AZ91D HP die cast parts, both abrasives produced some light etching using a 3-minute polish. No etching was observed on AM60 sections and very light etching was observed on thicker AZ91D sections. The preparation cycle developed from this work is given in Table 1.

The oil-base diamond suspensions must be cleaned from the specimens (and the fixture holding the specimens in an automated process) with alcohol or other suitable solvents. After each diamond- polishing step, the surface was scrubbed with cotton saturated with ethanol. Then, the surface was rinsed quickly with water, sprayed with alcohol, and dried with hot air. Exposure to water was always keep as brief as possible. This cleaning process can be used after the final step, or the specimens can be washed with a glycerol-ethanol solution. Cleaning without using water is inconvenient. Holding the specimen under running water for about a second eased the cleaning problem and did not appear to harm the microstructure. Cosmetic cotton puffs can scratch the surface when swab etching. If desired, a brief vibratory polish with one of the final abrasives could follow the practice. Longer times will produce etching.

A variety of standard etchants were used to reveal the microstructure. Table 2 lists these reagents. All are used by immersion. The aqueous hydrofluoric acid solution should be placed in a polyethylene beaker; otherwise glass can be used. The glycol etch and the acetic-glycol etch produced very similar results, but the later faintly reveals the grain boundaries. The acetic-picral etch also reveals the grain

boundaries lightly. These three etchants will outline the massive $Mg_{17}Al_{12}$ phase. The phospho-picral etch darkens the region around the massive phase, revealing it nicely by contrast. The aqueous HF etch darkens the massive phase preferentially.

Figure 1 shows the surface of an AZ91D HP die cast specimen after the 1-μm diamond step. Gas porosity (large round holes) and shrinkage cavities (irregular, narrow holes) are commonly observed in these castings. At the 1-μm stage, the surface is flat; there are no artifacts from preparation, such as comet tails, pull out, or smeared metal. Contra rotation is more aggressive than complementary and can promote comet tailing or excessive relief at very hard particles. If that is observed, repeat the last step using complementary rotation, and it will be eliminated. Only very fine scratches are present after this step.

Figures 2 and 3 show the as-polished surfaces of AZ91D and AM60 HP die cast specimens after the final step, using Masterprep alumina and Masterpolish suspensions, but before etching. The voids are properly revealed, without any artifacts, and only very fine scratches are visible. A few fine pits aligned in linear fashion can be seen when traversing the surface, but they are infrequent.

Figure 4 shows examples of mechanical twins observed in each alloy. The acetic-picral etch was the best for detecting these features. Figures 5 to 9 show the microstructures of both alloys revealed using the five etchants in Table 2.

The micrographs clearly show the structures that are always somewhat more complex for the AZ91D with its greater alloy content. With its higher aluminum content, AZ91D exhibits more segregation and contains more of the massive $Mg_{17}Al_{12}$ phase and more of the very fine precipitated phase, than AM60.

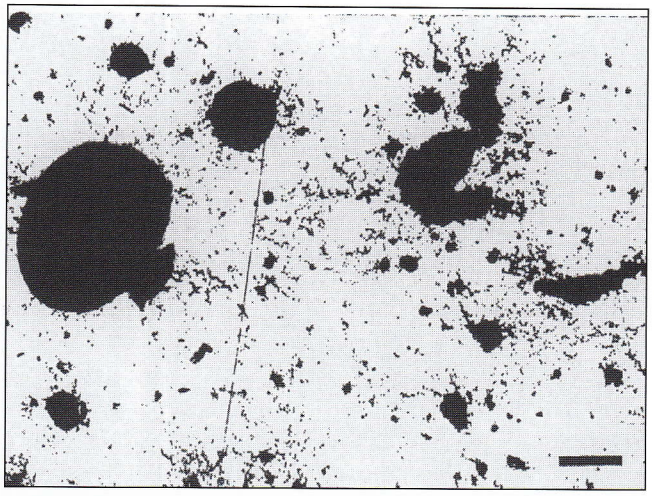

Figure 1. AZ91D surface after the 1-μm diamond step (as-polished) revealing numerous voids (magnification bar is 200-μm long).

Figura 1 - Superficie di AZ91D dopo il passaggio con diamante 1-μm che rivela numerosi vuoti (la barra di ingrandimento è lunga 200-μm)

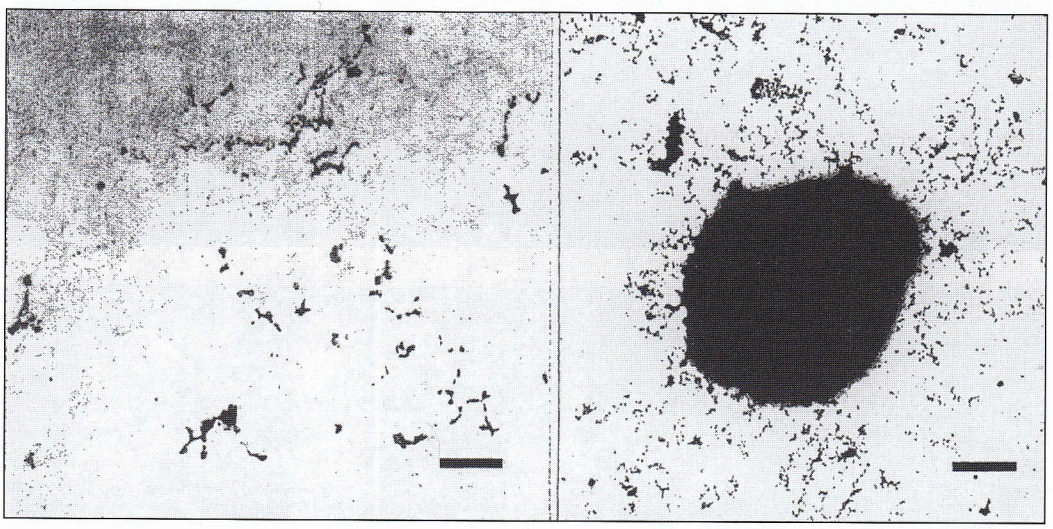

Figure 2a and b. As-Polished surfaces of AM60 (left) and AZ91D (right) alloys prepared using Masterprep alumina for the final step (magnification bars are 20- and 100-μm long, respectively).

Figura 2 - a) Superfici come lucidata di leghe AM60 e b) AZ91D preparate mediante Masterprep alumina per il passaggio finale (la barra di ingrandimento sono lunghe rispettivamente 20 e 100-μm)

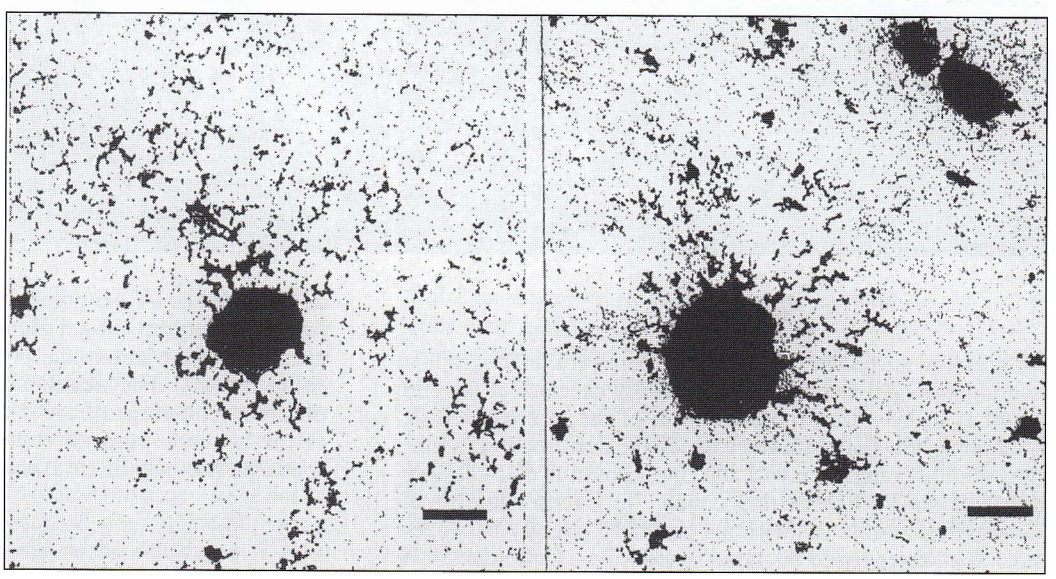

Figure 3a and b. As-Polished surfaces of AM60 (left) and AZ91D (right) alloys prepared using Masterpolish alumina-silica slurry for the final step (magnification bars are 100-μm long).

Figura 3 - a) Superfici come lucidata di leghe AM60 e b) AZ91D preparate mediante impasto Masterpolish alumina-silica per il passaggio finale (la barra di ingrandimento è lunga 100-μm)

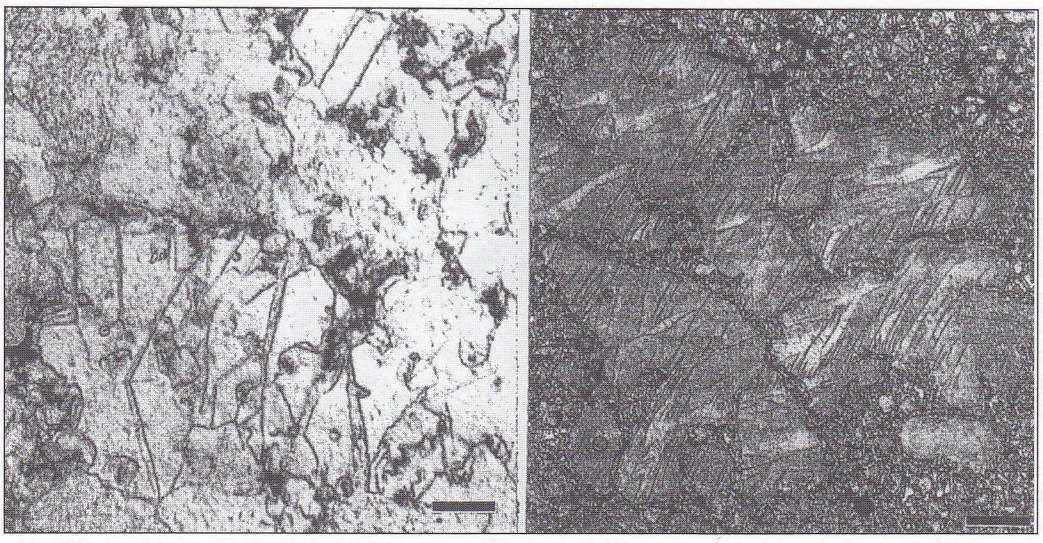

Figure 4a and b. Mechanical twinning observed in AM60 (left) and AZ91D (right) alloys prepared using Masterprep alumina for the final step and etched with acetic-picral (magnification bars are 10- and 20-μm long, respectively).

Figura 4 - Accoppiamento meccanico osservato in leghe a) AM60 e b) AZ91D dopo attacco chimico con agente picrico-acidico (la barra di ingrandimento sono lunghe rispettivamente 10-μm)

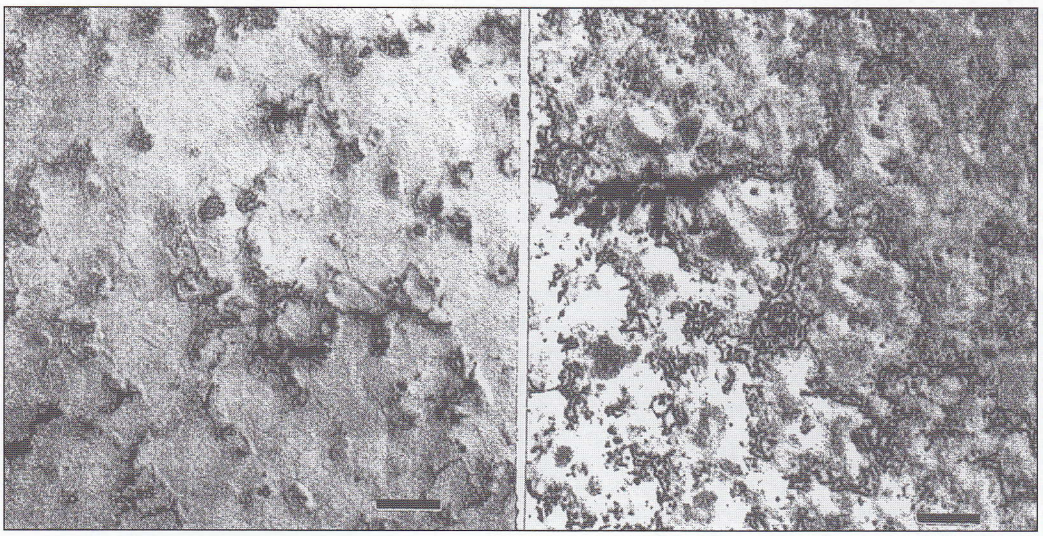

Figure 5a and b. Microstructures of AM60 (left) and AZ91D (right) alloys after etching with the glycol reagent (magnification bars are 10-μm long).

Figura 5 - Microstrutture di leghe a) AM60 e b) AZ91D dopo attacco chimico con reagente glicolico (la barra di ingrandimento sono lunghe rispettivamente 10-μm)

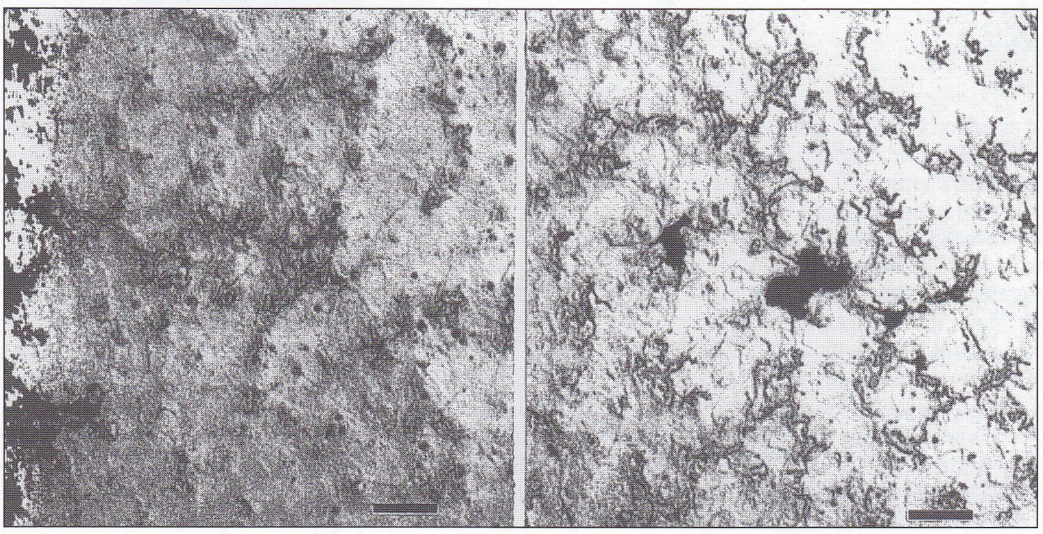

Figure 6a and b. Microstructures of AM60 (left) and AZ91D (right) alloys after etching with the acetic-glycol reagent (magnification bars are 10-μm long).

Figura 6 - Microstrutture di leghe a) AM60 e b) AZ91D dopo attacco chimico con reagente fosfo-picrico (la barra di ingrandimento sono lunghe rispettivamente 10-μm)

CONCLUSIONS

Magnesium alloys are difficult to prepare for metallographic examination. All steps must be carefully executed if the end result is to be a true representation of the microstructure. Sectioning must not introduce excessive deformation. Grinding should commence with the finest possible SiC abrasive that will remove the cutting damage in a reasonable time. Oil-based diamond suspensions produced slightly better results than water-based diamond suspensions. Colloidal silica can be used as a final polish for pure Mg but etches Mg alloys. Masterprep sol-gel alumina and Masterpolish, a mixture of alumina and silica, produced satisfactory final polishing results. The classic etchants do produce somewhat different renderings of the microstructure. Use of only one of the general- purpose etchants (glycol, acetic-glycol or acetic-picral) may be insufficient.

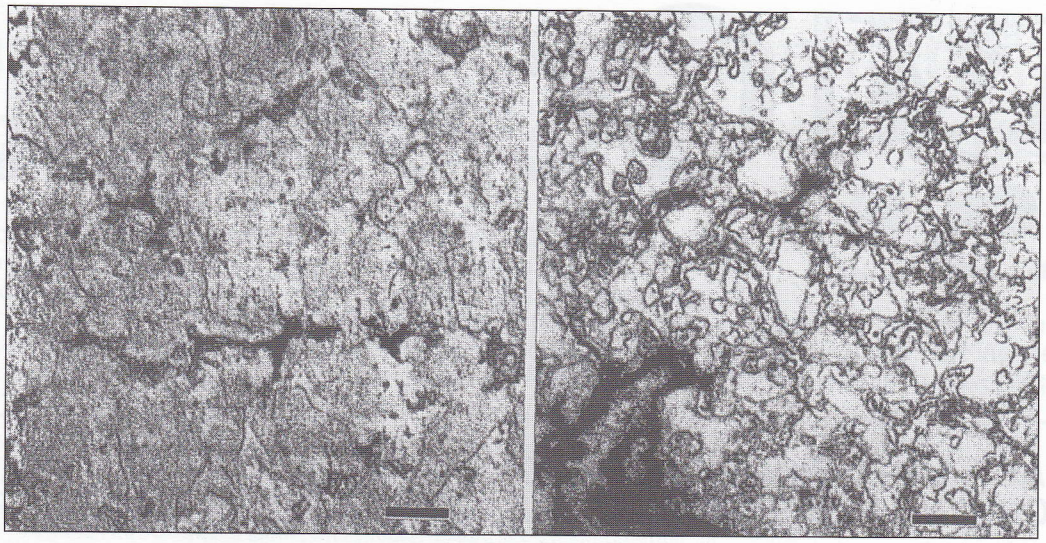

Figure 7a and b. Microstructures of AM60 (left) and AZ91D (right) alloys after etching with the acetic-picral reagent (magnification bars are 10-μm long).

Figura 7 - Microstrutture di leghe a) AM60 e b) AZ91D dopo attacco chimico con reagente 10% HF (la barra di ingrandimento sono lunghe rispettivamente 10-μm)

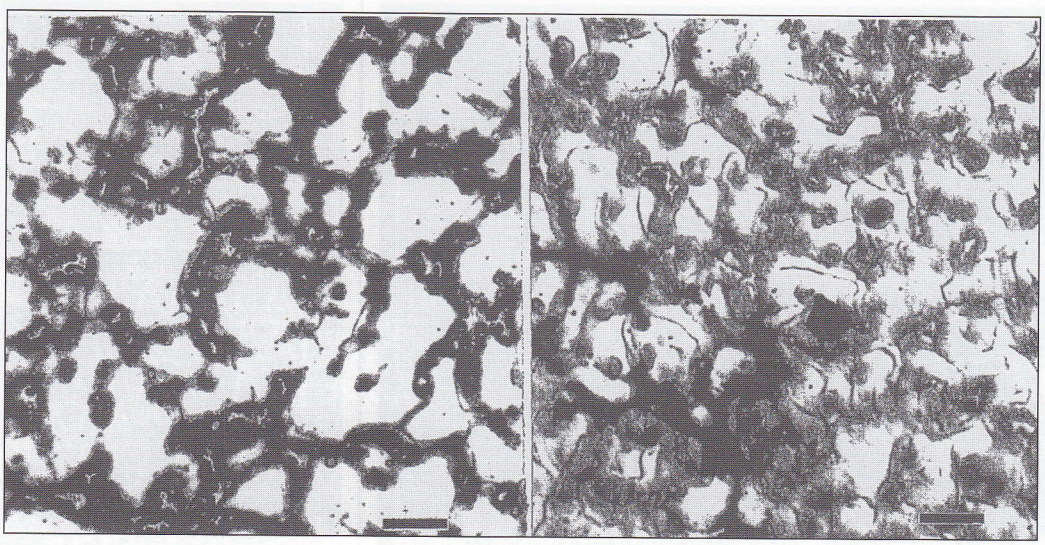

Figure 8a and b. Microstructures of AM60 (left) and AZ91D (right) alloys after etching with the phospho-picral reagent (magnification bars are 10-μm long).

Figura 8 - Microstrutture di leghe a) AM60 e b) AZ91D dopo attacco chimico con reagente fosfo-picrico (la barra di ingrandimento sono lunghe rispettivamente 10-μm)

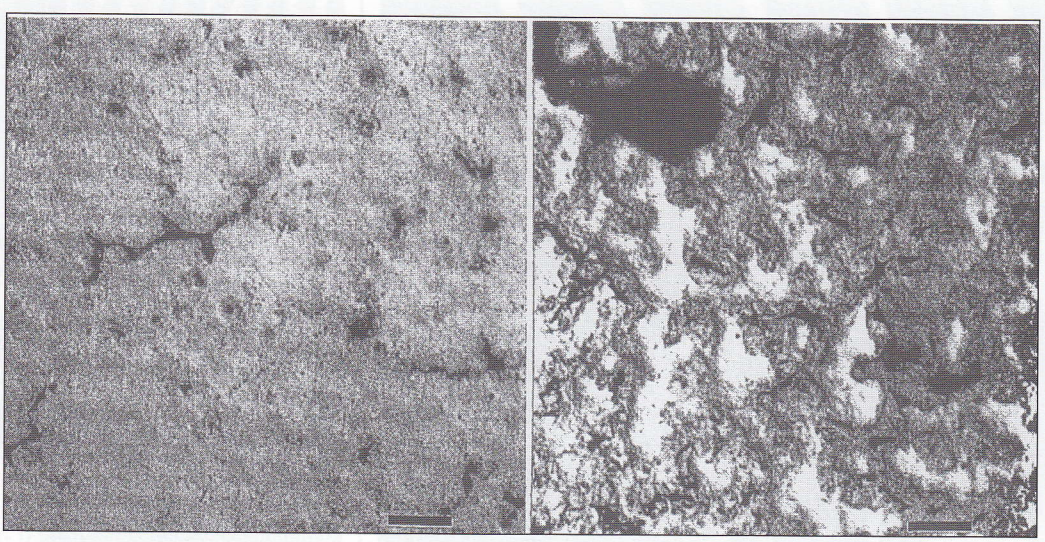

Figure 9a and b. Microstructures of AM60 (left) and AZ91D (right) alloys after etching with the 10% HF reagent (magnification bars are 10-μm long).

Figura 9a e b - Microstrutture di leghe a) AM60 e b) AZ91D dopo attacco chimico con reagente 10% HF (la barra di ingrandimento sono lunghe rispettivamente 10-μm)

METALLOGRAFIA DELLE LEGHE DI MAGNESIO PRESSOCOLATE

I campioni di magnesio e sue leghe, indipendentemente dai processi impiegati, sono fra i più difficili da preparare per l'effettuazione di esami microstrutturali. Il magnesio e le sue leghe hanno una scarsa durezza e resistenza ma contengono precipitati molto più duri. Ciò rende difficile l'eliminazione di graffi e deformazioni di matrice nonché il controllo del rilievo, ovvero l'eccessiva diversità di altezza fra matrice e precipitati. Il magnesio inoltre è molto reattivo e vi sono molte controversie sulla questione se l'acqua debba essere eliminata o meno dai passaggi della preparazione finale. Nonostante la sua struttura cristallina sia fitta e esagonale, Il magnesio non risponde bene alla luce polarizzata dopo la lucidatura.

Questa memoria presenta un nuovo metodo per preparare campioni di magnesio e sue leghe in grado di creare superfici lisce senza problemi di rilievi e graffi minori e deformazione di matrice. Vengono inoltre forniti commenti su agenti d'attacco.