ORIGINAL PAPER

STRUCTRUAL HARDENING MECHANISM OF LEAD-CADMIUM-BISMUTH-MAGNESIUM ALLOYS (PbCdBiMg) FOR BATTERY GRIDS FOR THE NEW GENERATION

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Abstract. *The phenomenon of aging and overaging of PbCdBiMg alloys is characterized by continuous and discontinuous transformation, it is hardening and softening characterized by a precipitation of cadmium. In general, the magnesium addition increases the hardness and delays the kinetics of the hardening processing. Indeed, the study of the return to equilibrium of supersaturated hardened alloys PbCdBiMg was carried out by various experimental techniques: hardness, micro hardness, optical microscopy and X-ray diffraction. Two structural states were considered: raw casting alloys and rehomogenized alloys. The alloys concerned are: Pb2% Cd1% Bi and Pb 2% Cd3% Bi with various magnesium proportions: 0.12% Mg, 1% Mg, 2% Mg and 3% Mg. The experimental temperatures are 20 ° C and 80 ° C. Bismuth accelerates the curing reaction and magnesium slightly retards the transformation.*

Keywords: *Alloy, Hardness, Optical Microscopy, X-Ray Diffraction.*

1. INTRODUCTION

The PbCdBi and PbCdBiSn alloys were studied systematically by Saissi and all in several studies [1, 2]. During aging, the two alloys undergo continuous and discontinuous transformations, while at the overaging; it is a discontinuous softening precipitation.

Due to their good mechanical properties, Pb-Mg alloys were recommended as a substitute for lead-tin alloys in sheathing for telephone cables [3]. Indeed, hardening the Pb-Mg alloy showed a hardness evolution since it evolved from 14.1 Brinell to 19.1 Brinell after quenching after 1 day of holding at room temperature for a composition of 3.5 % Mg.

Table 1 includes values of different hardness that the Pb-Mg alloy peaked for various compositions:

Table 1. Hardening of lead-magnesium alloys [3]

Magnesium in Wt %	At cast conditions			
	0.2	0.5	2.5	3.5
Brinell Hardness kg/mm^2 at once	5.8	9.75	10.7	14.1
Brinell hardness kg/mm^2 after 1 day	5.9	9.5	15.3	19.5

Hence the idea to add Magnesium to our master alloys Pb-Bi-Cd according to defined proportions. Our goal is to study systematically the aging and overaging processes for soaked

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supersaturated solutions of these alloys by different techniques: hardness, optical microscopy and X-ray diffraction.

2. EXPERIMENTAL TECHNIQUES

2.1. Preparation of alloys

The alloys are prepared from pure metals: lead (99.99%), cadmium (99.99%), bismuth (99.99%) and magnesium (99.99%).

The phase diagram of Pb-Mg alloy according to Figures 1 and 2 shows a eutectic at 248 ° C with a composition of 2.25% Mg. The maximum solubility of magnesium in the lead is 0.75% Mg. A 19% Mg solid solution Mg_2Pb appears.

Jänecke [3] carefully investigated the structure of this ternary system, in order to throw some light on the binary magnesium-cadmium system. Only the lead corner of the ternary system is presented here, the atomic percentages given by Jänecke [3] being converted into weight percentages. The lead corner has a ternary eutectic at the composition 18.9 Wt% Cd, 1.8 Wt% Mg, 79.3 Wt% Pb, and temperature 194°C. It corresponds to the reaction: E_T ~ Lead solid solution + Cadmium solid solution + Mg_2Pb.

The weight concentrations of magnesium chosen for the development of these alloys are: 0.12% Mg, 1% Mg, Mg 2%, 3% Mg.

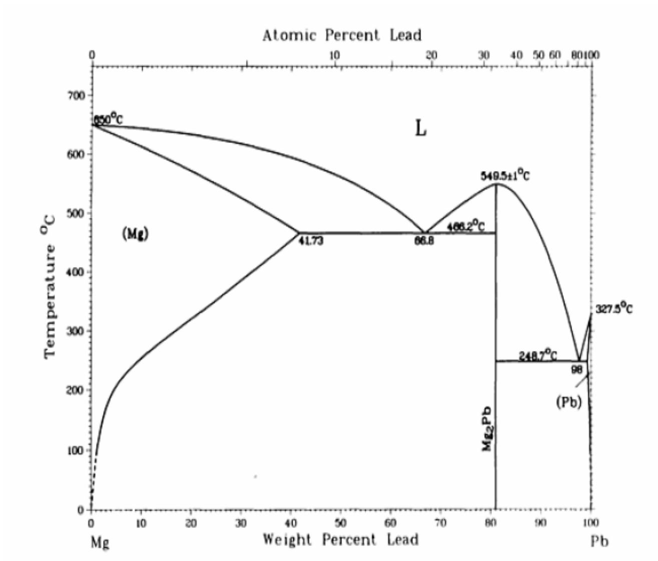

Figure 1. Pb-Mg Phase diagram [4].

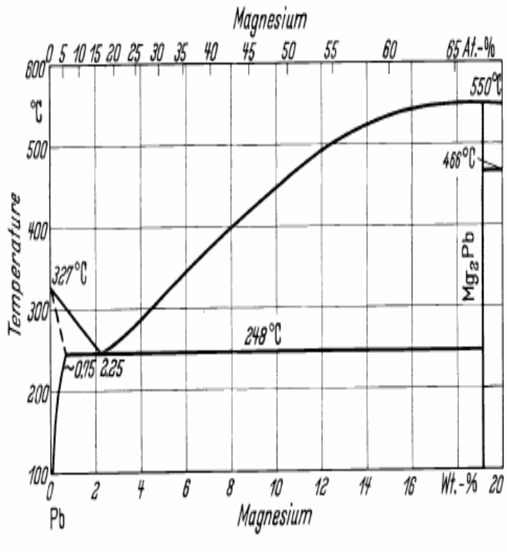

Figure 2. Pb-Mg phase diagram (according to Horsley and Hansen) [3].

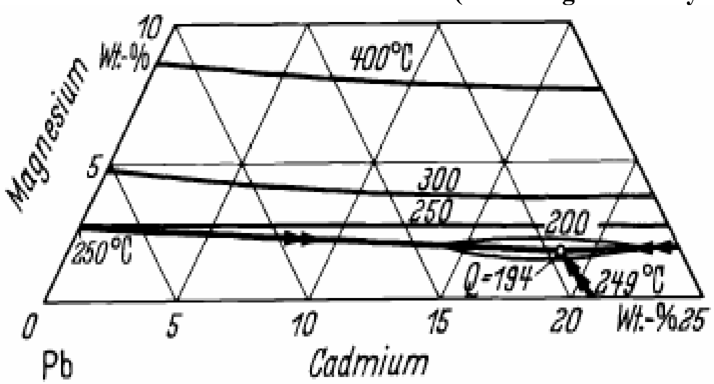

Figure 3. Lead-Cadmium-Magnesium phase diagram according to Janecke [3].

We developed alloys whose compositions are shown in Table 2.

Table 2. Composition of the studied alloys.

Content by weight (%Cd)	Content by weight (%Bi)	Content by weight (%Mg)
2	1	0,12
		1
		2
		3
2	3	0.12
		1
		2
		3

To study the structural-cast alloys, the elements taken in the proper proportions, are introduced into a silica ampoule of 8 mm in diameter, sealed under high vacuum, the mixture is heated to 500°C. After melting and cooling total, the tube is quenched with water. The samples were examined directly or may be stored in liquid nitrogen. For rehomogenization, the ingot obtained is cut into several pieces that are then polished by abrasion. Samples are introduced into silica ampoules sealed under high vacuum. The whole is kept at 264 °C for 2 h (estimated optimal for rehomogenization) and then quenched with water.

2.2. Hardness

The hardness tests are carried out by the □ickers method, using a durometer testweel under a load of 2 kgf. Each measurement is the average of up to five trucks spread over a flat section corresponds to a diametric plane or perpendicular to the axis of the cylindrical sample. The sections are obtained by sawing, mechanical abrasion and chemical polishing. Recall that the empirical relation HV= 0.3 (Mpa) can be used to evaluate the rupture strength ® of these alloys.

2.3. Technical micrographic observation

The physical properties of solid solutions for soaked lead alloys change from room temperature. The curing mechanisms correspond to continuous and/or discontinuous transformations. Indeed, this temperature corresponds to 0.5 T_{fusion} (°K), temperature at which the alloy elements can diffuse.

The observation of the structure of alloys corresponding to the continuous/discontinuous transformations is made by using the optical microscope, the samples must undergo mechanical polishing, chemical polishing using hydrogen peroxide 30% H_2O_2 and three share of glacial acetic acid for 20 seconds to 2 minutes, then chemical attack using 100g of ammonium molybdate; 250g citric acid and water in sufficient quantity to have a liter of the mixture [5].

3. RESULTS

3.1. STUDY OF THE ALLOY Pb2%Cd1%Bi2%Mg:

3.1.1. Hardness

Fig. 4 shows the change in hardness with time at 20°C of raw casting alloys Pb 2%Cd1% Bi [1], and Pb 2%Cd1% Bi 2% Mg. Both curves have the same shape, but we note that the effect of magnesium is manifested by an increase in hardness and a delay of the hardening reaction. Indeed in the absence of magnesium, hardness is about 11.125 HV after 6 minutes and culminates 12.53 HV (maximum) after 80 minutes of aging.

In the presence of magnesium (2% by weight) the hardness is 12.835 HV after 10 minutes and the maximum (approximately 13.754 HV) is obtained until after 154 minutes.

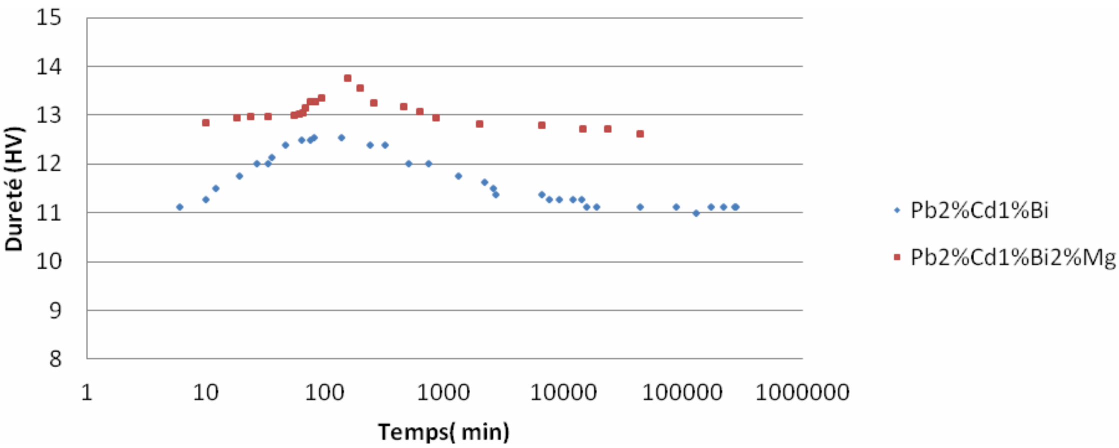

Figure 4. Evolution of hardness versus time at 20 ° C for as-cast alloys Pb2%Cd1%Bi and Pb2%Cd1%Bi2%Mg.

3.1.2. Evolution of quenching structure of the as-cast alloy Pb 2% Cd1% Bi2%Mg:

The return to equilibrium of the quench structure can be visualized by metallographic observations. Figure 5 shows the discontinuous processing of the alloy Pb2% Bi2% Cd1% Mg after 30 min of quenching during aging.

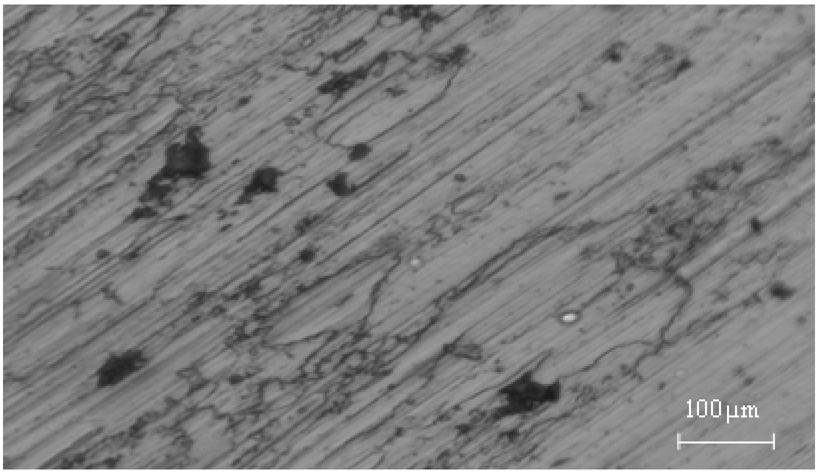

Figure 5. Visualization of the discontinuous transformation of Pb2%Cd 1%Bi 2% Mg as-cast alloy quenched with water at 20 ° C during aging after 20 min quenching.

Indeed, when the increase in hardness is accompanied by the displacement of the fronts, this is a discontinuous processing. Otherwise, it is a continuous reaction. Figure 6 shows the progress of the reaction fronts of a discontinuous processing. This reaction is partial and its kinetic is increasingly slow, which makes it very incomplete.

After 65 minutes of tempering, was achieved microhardness measurements and it found that areas transformed by the discontinuous reaction are less hard than continuous reaction, this shows that the metallographic observations and microhardness tests are according to the hardness measurements.

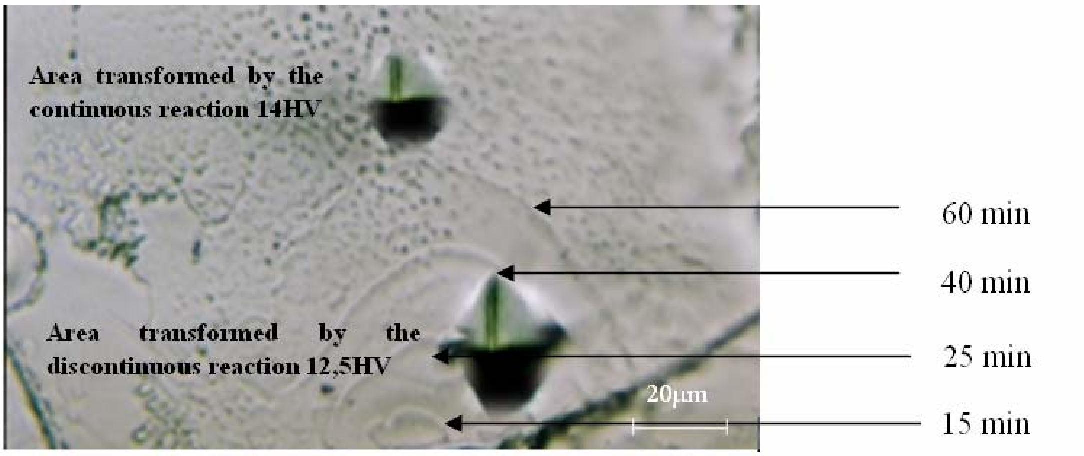

Figure 6. a) Visualization of displacement of grain boundaries after successive chemical attacks after quenching at 20 ° C of the crude casting alloy Pb2% Cd1% Bi2% Mg for 15 min, 25 min, 40 min and 60 min; b) Visualization of footprints of micro hardness performed on the crude casting alloy Pb2% Cd1% Bi2% Mg aged 65 minutes at 20 ° C.

Figure 7 shows another typical example of the transformations sites obtained from crude casting samples aged and chemically attacked several times at room temperature. The germ growth occurs after a time of approximately 15 minutes of aging.

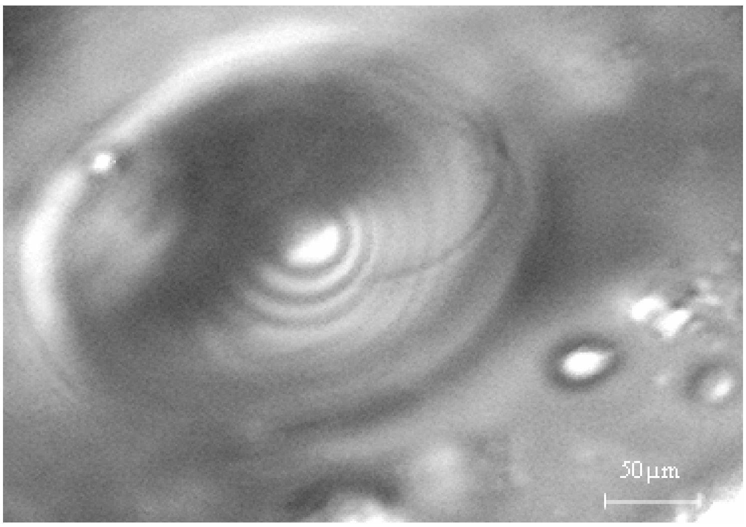

Figure 7. Visualization of Grain boundaries displacement of the crude casting alloy Pb 2% Cd 1% Bi 2%Mg tempered with water at 20 ° C during aging after 15 min of quenching after 5 successive attacks.

Stand for prolonged time, there overaging. It is manifested by discontinuous precipitation that allows the formation of large precipitates type Cd. This reaction is softening as confirmed by hardness measurements performed on this sample maintained at 20°C after 3 days of hardening (Fig. 8).

Figure 8. Visualization of discontinuous transformation of crude casting alloy Pb 2% Cd 1% Bi 2%Mg tempered with water at 20°C during overaging after 3 days of quenching.

3.2. INFLUENCE OF HOLDING TEMPERATURE (80 °C) OF THE ALLOY Pb2% Cd 1% Bi 2% Mg

3.2.1. Hardness

Figure 9 shows the change in hardness with time at temperatures 20°C and 80°C of the raw casting alloy Pb2%Cd1%Bi 2% Mg. The supersaturated tempered alloy has a hardness of 12.835 HV which is almost twice that of pure lead whose hardness varies between 4 HV and 5 HV, which proves that the alloy is partially transformed during cooling. At 20°C; the maximum hardness is 13.754 HV and is reached after 154 minutes, then the hardness slightly decreased to 12.62 HV beyond one month. At 80 ° C, the maximum hardening is reached after 108 min (~ 14 HV), the hardness decreases sharply to peak 12HV after 4 weeks of quenching during overaging.

Furthermore, the influence of temperature 80°C being the temperature of ripening of battery plates and the highest temperature of battery operation results in acceleration of hardening reaction and an increase in hardness.

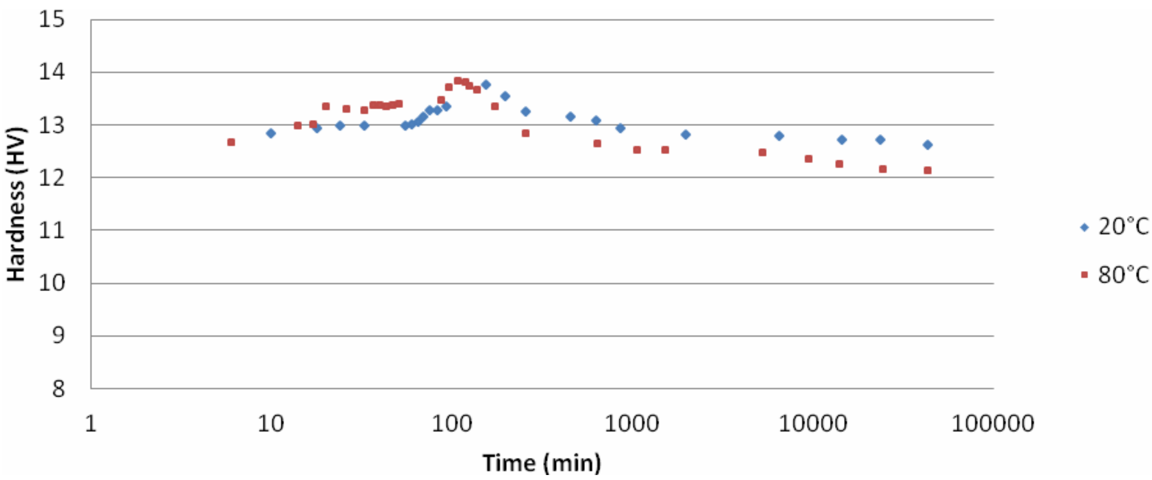

Figure 9. Evolution of the hardness of the crude casting alloy Pb2% Cd1% Bi2%Mg at temperatures 20°C and 80°C versus time.

3.2.2. Evolution of quenching structure of the alloy Pb 2% Cd1% Bi2%Mg

The structure of the alloy Pb2 %Cd1% Bi 2% Mg aged around 80°C appears similar to that of 20 °C, this has been proven by hardness measurements and optical microscopy. Indeed, its witness the same structural phenomena; Ageing is characterized by a batch processing and continuous hardening reaction, while the over-aging is characterized by a softening discontinuous transformation.

Figure 10 illustrates the discontinuous processing of the alloy Pb2 %Cd1% Bi 2% Mg maintained to 80°C during aging, while Figures 11 and 12 visualize the same transformations during overaging. Effectively, gradually, as we advance in time, we are witnessing the formation of large precipitates.

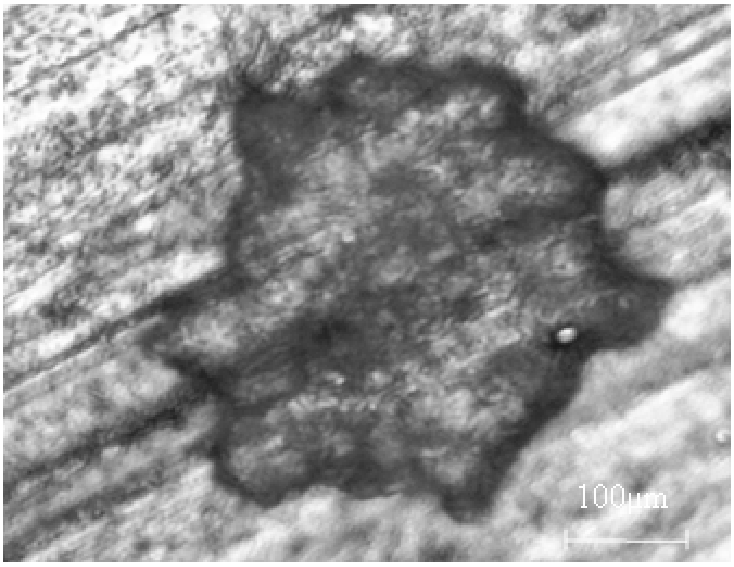

Figure 10. View of the discontinuous processing of the alloy Pb 2%Cd 1% Bi2% Mg maintained at 80°C during aging after 45 min quenching.

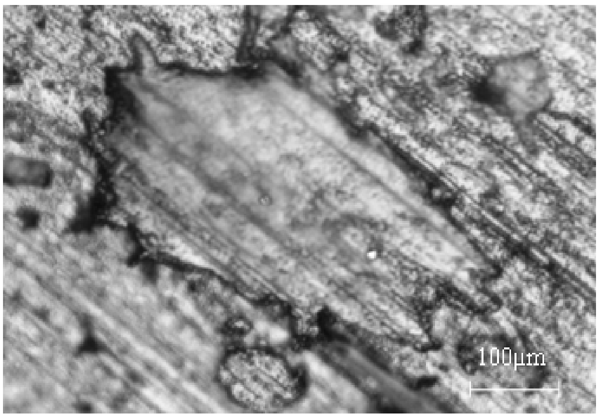

Figure 11. Visualization of the discontinuous processing of the alloy Pb2% Cd1% Bi2% Mg maintained at 80 ° C during overaging after 4 hours of quenching.

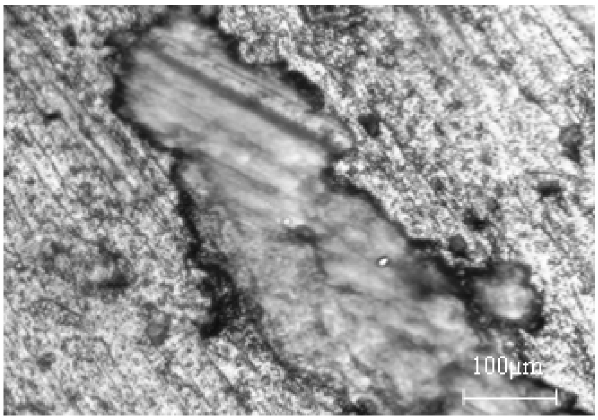

Figure 12. Visualization of the discontinuous processing of the alloy Pb2% Cd1% Bi2% Mg maintained at 80 °C during overaging after 2 days of quenching.

Figure 13 receives the eutectic mixture of this alloy during aging; figure 14 shows that the matrix becomes swept by the discontinuous processing which proves the decrease in hardness.

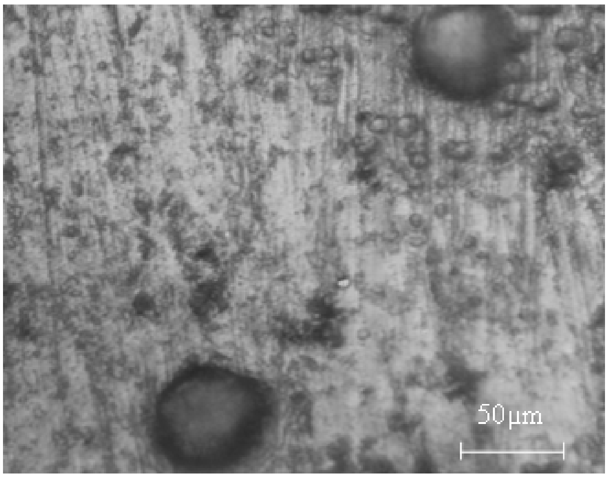

Figure 13. Visualization of the discontinuous processing of the alloy Pb2% Cd3% Bi2% Mg maintained at 20°C during overaging after 4 days of quenching.

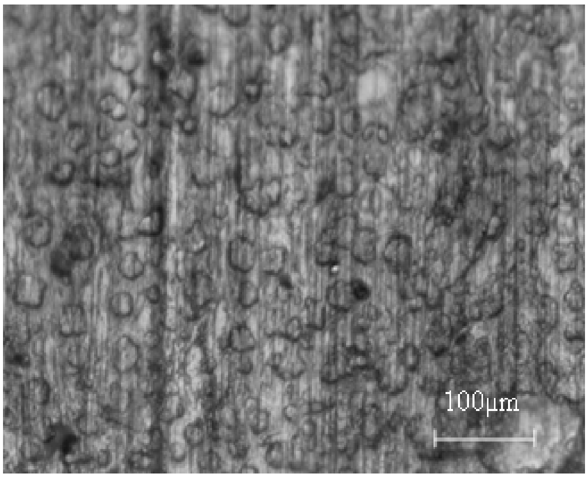

Figure 14. Visualization of the discontinuous processing of the alloy Pb2% Cd3% Bi2% Mg maintained at 20°C during overaging after 1 month of quenching.

3.3. INFLUENCE OF MAGNESIUM

To study the influence of magnesium on the hardening mechanisms of PbCdBiMg alloys, we worked on four different compositions. We selected the alloy whose hardness is maximum referring to Saissi and all [1], it is the Pb2% Cd3% Bi. It was obtained the hardness curves (Fig. 15).

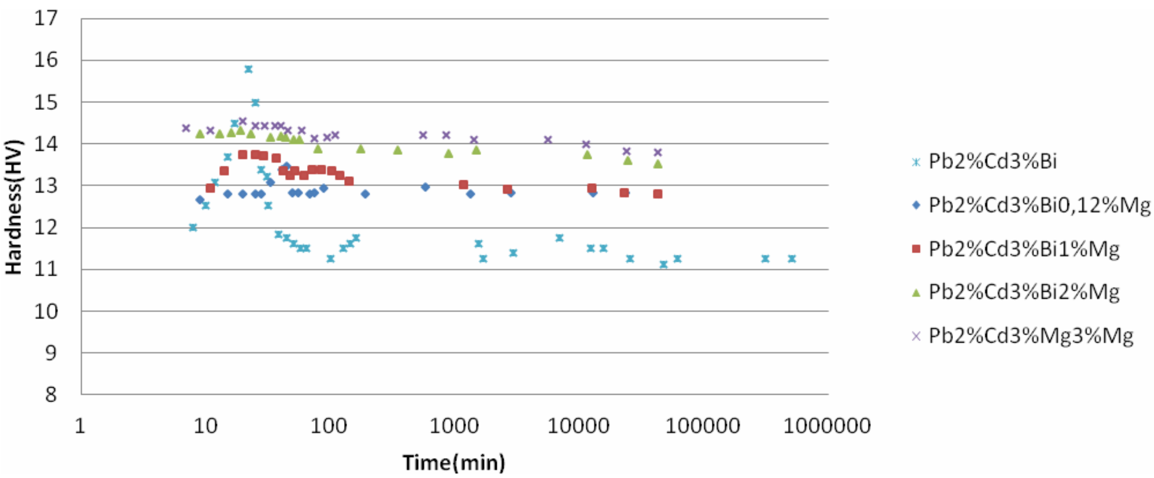

Figure 15. Evolution of hardness versus time at room temperature of the crude casting alloys Pb2%Cd3%Bi ; Pb2%Cd3%Bi0, 12%Mg , Pb2%Cd3%Bi1%Mg, Pb2%Cd3%Bi2%Mg and Pb2%Cd3%Bi3%Mg.

According to figure 15, the magnesium slightly increases the hardness but the kinetics of the hardening reaction is not clear, this is probably due to the presence of a high concentration of bismuth since it is an accelerator which is always consistent with the results found previously [1] and [6].

In addition, bismuth accelerates the hardening reaction and slightly increases the hardness, magnesium also increases hardness. During overaging we see that there is a stagnation of hardness after one day holding with a slight increase in hardness of about 1 HV (Fig. 15).

To further visualize the influence of magnesium we decreased the percentage of bismuth, and studied the alloy Pb2% Cd1% Bi. The results are shown in figure 16.

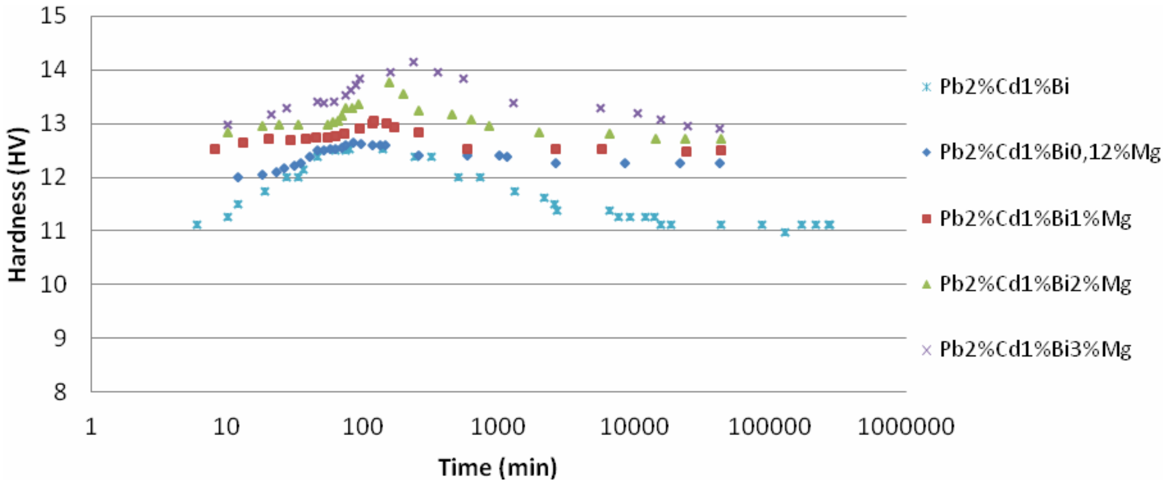

Figure 16. Evolution of hardness versus time at room temperature of the crude casting alloys Pb2%Cd1%Bi; Pb2%Cd1%Bi0, 12%Mg , Pb2%Cd1%Bi1%Mg, Pb2%Cd1%Bi2%Mg and Pb2%Cd1%Bi3%Mg.

From figure 16 and with low bismuth content were able to visualize the effect of magnesium and those by working on four different compositions (0.12% Mg, 1% Mg, 2% and 3% Mg).

Indeed, as the magnesium concentration increases, the aging process seems to be more delayed and mechanical properties improve in terms of hardness, this is proved by metallographic observations for these types of alloys as shown in figures 17-24.

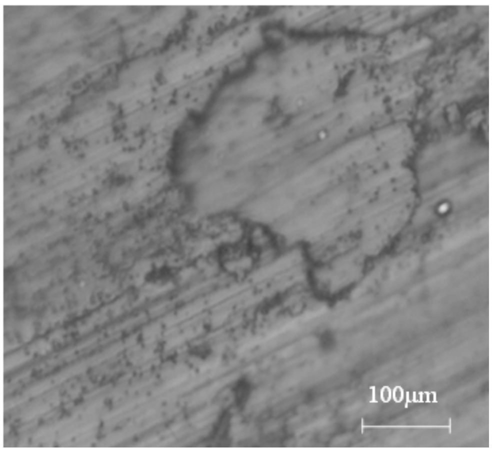

Figure 17. Visualization of the discontinuous processing of the alloy Pb2%Cd1% Bi 0, 12% Mg maintained at 20°C during aging after 15 min of quenching.

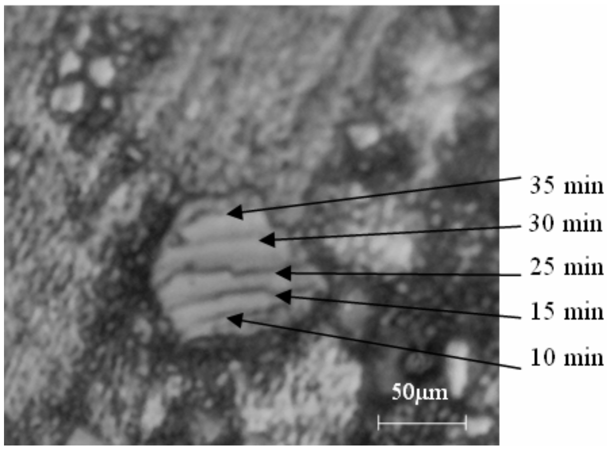

Figure 18. Visualization of displacement of grain boundaries after successive chemical attacks after quenching at 20 °C of the crude casting alloy Pb2% Cd3% Bi 0, 12% Mg for 10 min, 15 min, 25 min, 30 min and 35 min.

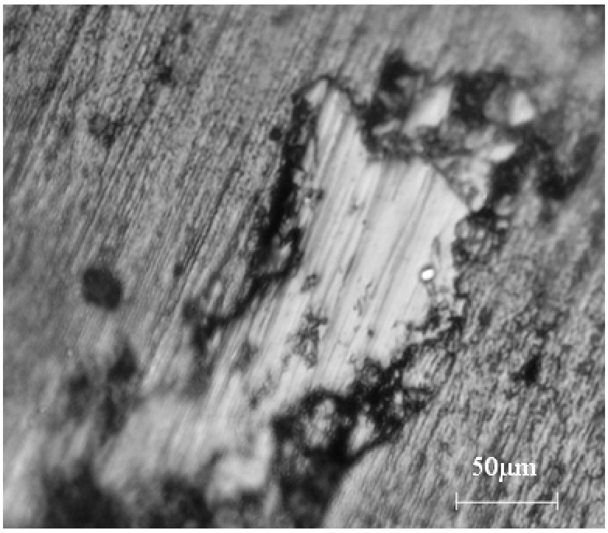

Figure 19. Visualization of the discontinuous processing of the alloy Pb2%Cd3% Bi 0, 12% Mg maintained at 20°C during overaging after 1 hour of quenching.

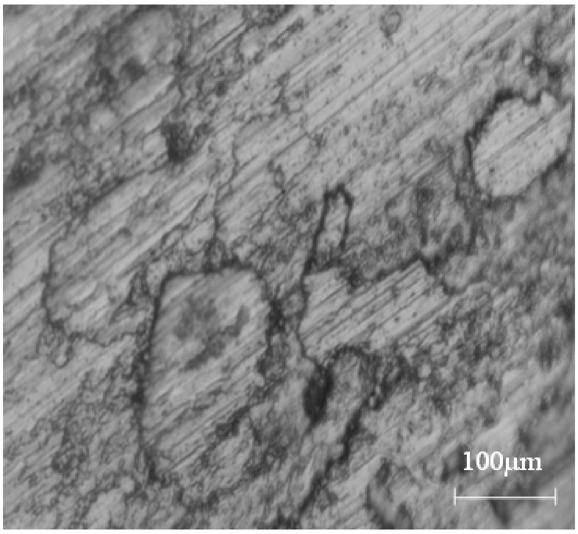

Figure 20. Visualization of the discontinuous processing of the alloy Pb2%Cd1% Bi 0, 12% Mg maintained at 20°C during aging after 2 hours 30 min of quenching.

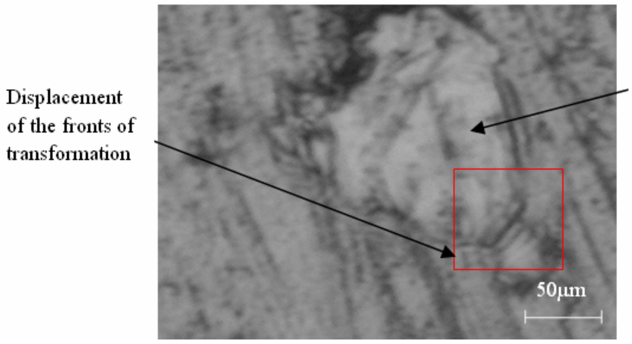

Figure 21. Visualization of the discontinuous processing of the alloy Pb2%Cd1%Bi1%Mg maintained at 20°C during aging after 30 min of quenching.

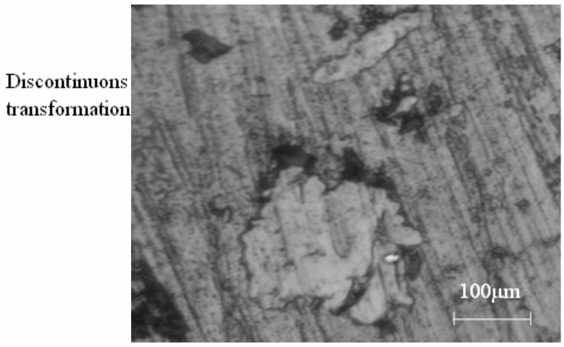

Figure 22. Visualization of the discontinuous processing of the alloy Pb2%Cd1%Bi1%Mg maintained at 20°C during overaging after 1 day of quenching.

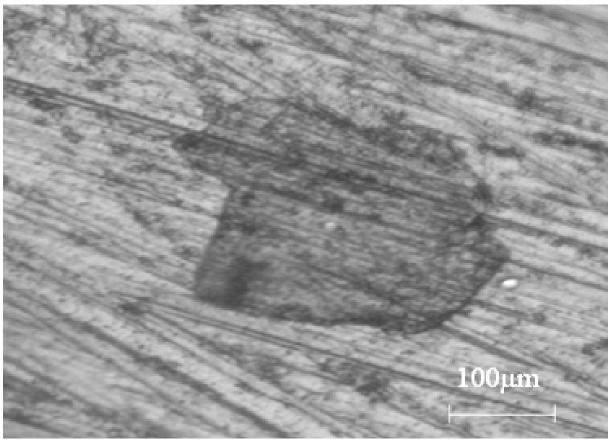

Figure 23. Visualization of the discontinuous processing of the alloy Pb2%Cd3%Bi3%Mg maintained at 20°C during aging after 67 min of quenching.

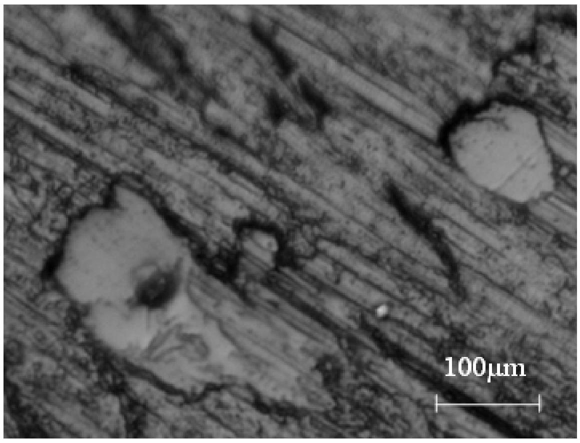

Figure 24. Visualization of the discontinuous processing of the alloy Pb2%Cd3%Bi3%Mg maintained at 20°C during overaging after 3 days of quenching.

3.4. INFLUENCE OF REHOMOGENIZATION

Figure 25 shows the evolution of the hardness as a function of time at room temperature of raw casting alloys rehomogenized and quenched with water. Generally the rehomogenized alloys age in the same way as the raw casting except that there is an acceleration of the hardening reaction for those rehomogenized. Indeed, treatment of rehomogenization reduces diffusion heterogeneities composition due to segregation phenomena which appear in the solidification structure leaving understand that reducing initiation sites of the discontinuous transformation amplifies hardness. The maximum hardness of rehomogenized samples (14.03HV) is higher than that of the raw casting samples (13.75HV). This is due to the dissolution of segregated phase during the treatment of rehomogenization, which increases the supersaturation after quenching at 20°C.

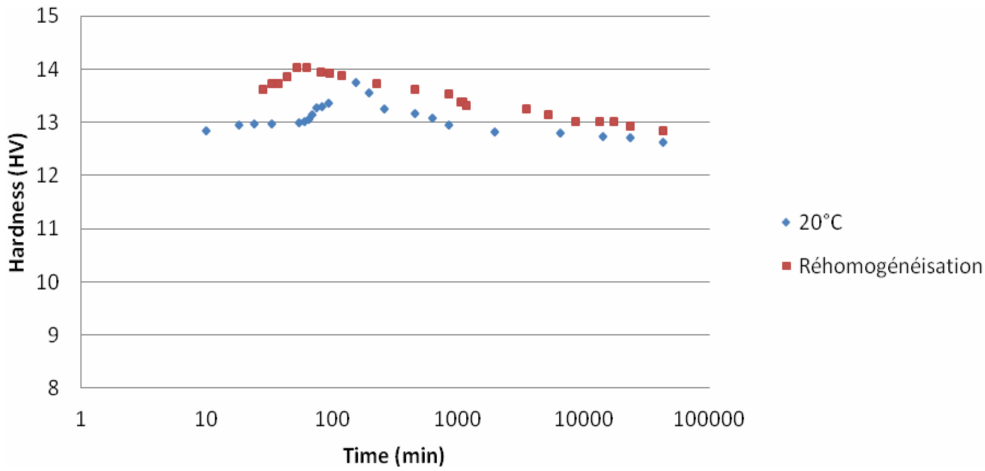

Figure 1. Evolution of hardness as function of time at 20°C, for alloy Pb2%Cd1%Bi2%Mg, as-cast and rehomogenized and then soaked in water.

Figures 26 and 27 show the same discontinuous transformations of raw casting alloys. Similarly, figures 28 and 29 put the item on eutectic mixtures that we identified for alloys kept at 20°C. Overall, Microscopic observations show that the structural hardening mechanisms of rehomogenized alloy are similar to those of the as-cast alloy, the aging effect is characterized by a discontinuous processing and a hardening continues reaction and overaging is characterized by softening discontinuous precipitation.

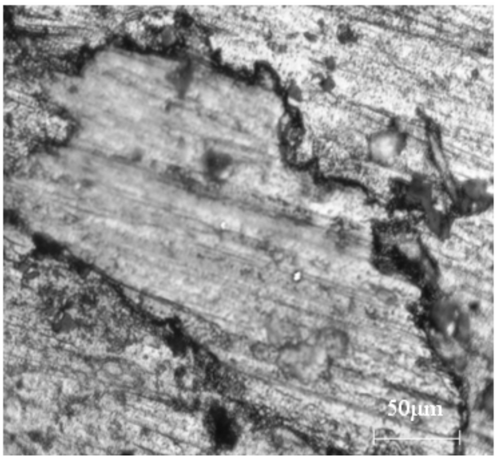

Figure 26. Visualization of the discontinuous processing of rehomogenized alloy Pb2%Cd1%Bi2%Mg maintained at 20°C during aging after 20 min of quenching.

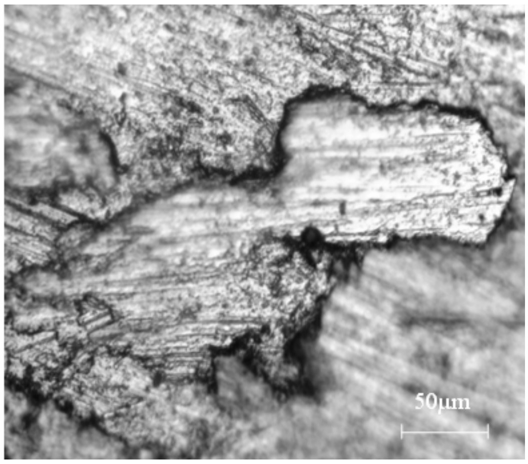

Figure 27. Visualization of the discontinuous processing of rehomogenized alloy Pb2%Cd1%Bi2%Mg maintained at 20°C during aging after 37 min of quenching.

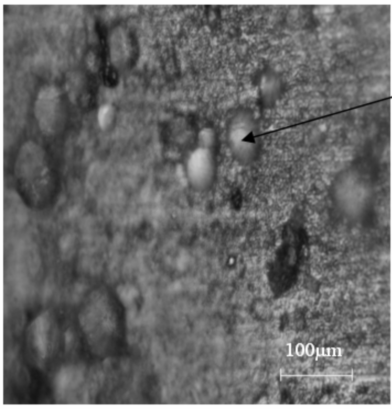

Figure 28. Visualization of the discontinuous transformation characterized by eutectic mixtures of Pb2%Cd 3% Bi2%Mg tempered and rehomogenized in water at 20°C during aging after 25 min quenching.

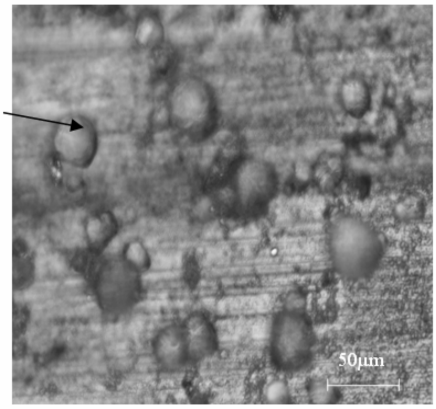

Figure 29. Visualization of the discontinuous transformation characterized by eutectic mixtures of Pb2%Cd 3% Bi2%Mg tempered and rehomogenized in water at 20°C during aging after 30 min quenching.

4. X-RAY DIFFRACTION

4.1. RESULTS INTERPRETATION OF THE XRD ANALYSIS FOR THE SAMPLE Pb2%Cd1%Bi0.12%Mg

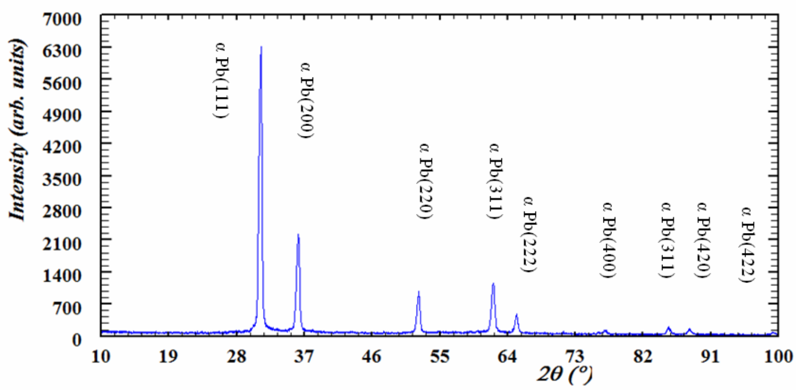

Figure 30. X-ray diffraction analysis of the surface of the alloy Pb2%Cd1%Bi 0.12%Mg.

According to the X-ray diffraction diagram of figure 30, the processing of diffractograms or spectra of the alloy Pb2% Bi Cd1% 0.12% Mg, highlights the peaks of the solid solution α lead, with a CFCs crystalline structure.

Table 3. Crystallographic parameters of the alloy Pb2% Cd1% Bi 0. 12% Mg

2θ [7]	2θ Calculated	d_{hkl}	hkl	Parameter a
31.270	30.86299	2.88635	111	4.9993
36.262	36.00106	2.50398	200	5.0079
52.220	52.13224	1.77110	220	5.0094
62.139	62.04992	1.51620	311	5.0286
65.233	65.27615	1.44482	222	5.0050
76.985	76.86670	1.25682	400	5.0272
85.416	85.35051	1.14530	331	4.9922
88.189	88.09879	1.11662	420	4.9936
99.334	99.21136	1.01784	422	4.9863

4.2. INTERPRETATION OF THE RESULTS OF THE XRD ANALYSIS FOR THE SAMPLE Pb2%Cd1%Bi3%Mg

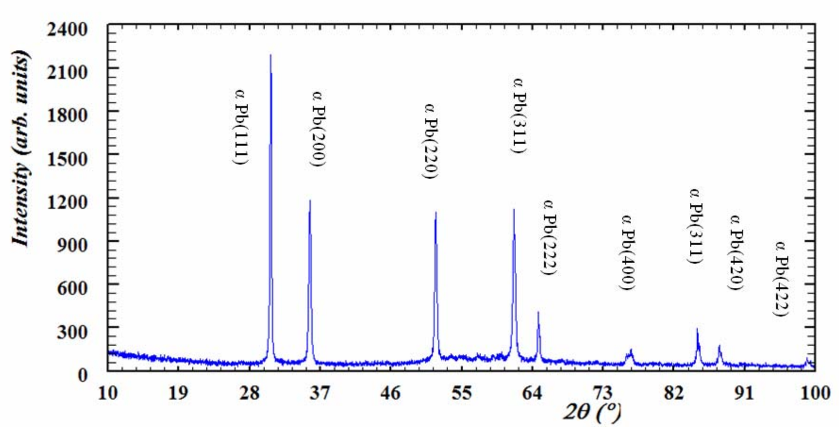

Figure 31. X-ray diffraction analysis of the surface of the alloy Pb2%Cd1%Bi 3%Mg.
According to the X-ray diffraction diagram of figure 31, the processing of diffractograms or spectra of the alloy Pb2%Cd1%Bi 3%Mg, highlights the peaks of the solid solution α lead, with a CFCs crystalline structure.

Table 4. Crystallographic parameters of the alloy Pb2% Cd1% Bi 3% Mg

2θ [7]	2θ Calculate	d_{hkl}	hkl	Parameter a
31.270	30.862	2.928	111	5.071
36.262	35.763	2.546	200	5.092
52.220	51.654	1.781	220	5.037
62.139	61.571	1.510	311	5.008
65.233	64.678	1.451	222	5.026
76.985	76.388	1.259	400	5.036
85.416	84.992	1.148	331	5.004
88.189	87.859	1.114	420	4.981
99.334	99.091	1.014	422	4.967

4.3. INTERPRETATION OF THE RESULTS OF THE XRD ANALYSIS FOR THE SAMPLE Pb2%Cd3%Bi2%Mg

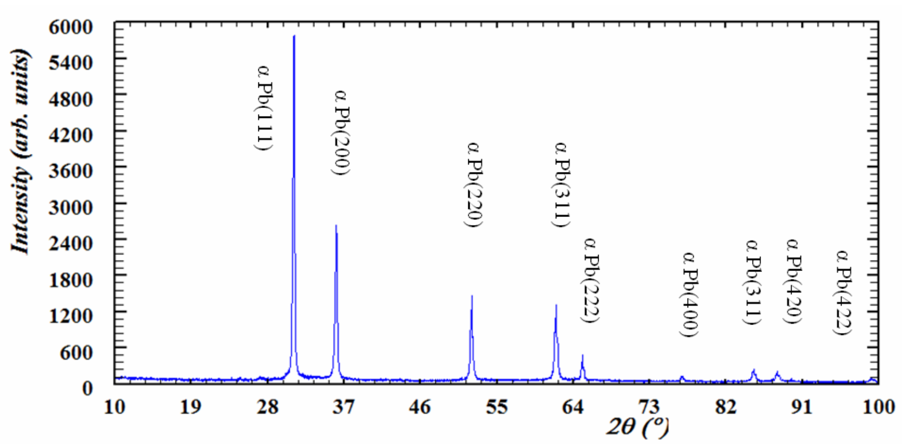

Figure 32. X-ray diffraction analysis of the surface of the alloy Pb2%Cd3%Bi 2%Mg.
According to the X-ray diffraction diagram of figure 32, the processing of diffractograms or spectra of the alloy Pb2%Cd3%Bi 2%Mg, highlights the peaks of the solid solution α lead, with a CFCs crystalline structure.

Table 5. Crystallographic parameters of the alloy Pb2% Cd3% Bi 2% Mg

2θ [7]	2θ Calculate	d_{hkl}	hkl	Parameter a
31.270	30.982	2.918	111	5.054
36.262	36.001	2.514	200	5.028
52.220	52.012	1.772	220	5.012
62.139	61.930	1.509	311	5.004
65.233	64.917	1.445	222	5.005
76.985	76.802	1.252	400	5.008
85.416	85.083	1.151	331	5.017
88.189	87.987	1.118	420	4.999
99.334	99.064	1.017	422	4.982

Interpretation

The diagrams of X-ray diffraction of the alloys Pb2%Cd1%Bi0,12%Mg, Pb2%Cd1%Bi3%Mg and Pb2%Cd3%Bi2%Mg (Figs. 30 - 32), visualize only the peaks of the solid solution α lead, which crystal structure is a cubic face-centered.

However, we can not say that the precipitation of secondary phases has not happened; the deduction we can make is that the X ray diffraction could not bring out such precipitation and those; seen their small size compared to that of lead. This is consistent with the results found earlier by Saissi and all [2].

4. CONCLUSIONS

The structural hardening mechanisms of Pb-Cd-Bi-Mg alloy faces the same phenomena as the alloys Pb-Bi-Cd [1] and Pb- Bi-Cd-Sn [2] already studied. Indeed, aging is characterized by a continuous reaction and discontinuous transformation while the overaging is characterized by softening discontinuous precipitation.

The alloys containing magnesium showed a hardness development compared to those free of magnesium. Indeed, as and when the magnesium content increases the hardness increases further and the kinetics of the hardening reaction occurs slowly. However, bismuth being an accelerator of the hardening reaction is in competition with magnesium, as the case of tin, which influences the speed of aging.

The dissolution of segregated phases in the treatment of rehomogenization increases the supersaturation after quenching by eliminating segregation cells outcome from solidification structure. Indeed, the hardening process for rehomogenized alloys are identical to those of the raw casting alloys but the kinetics of aging appear more accelerated, resulting in an increase in hardness of approximately 1 HV.

Moreover; discontinuous transformations of raw casting alloys and those rehomogenized are also characterized by eutectic mixtures.

Besides lead, the results of analyzes by X-ray diffraction of the alloys Pb2%Cd1%Bi0,12%Mg, Pb2%Cd1%Bi3%Mg and Pb2%Cd3%Bi2%Mg, were not able to show the other elements of the alloy, this is due to their very low compositions.

Finally, comparing the hardness values obtained by alloys PbCdBiMg to those given by the ternary alloys PbCdBi shows the beneficial effect of magnesium additions upon the mechanical properties of these alloys. Indeed, given that bismuth is an accelerator, we could not know the maximum hardness for the alloys: Pb2%Cd3%Bi0, 12%Mg, 1%Mg, 2%Mg and 3%Mg, knowing that this same alloy without Mg peake 16 HV. But it is strongly observed that all of the maximum hardness values obtained were higher in the presence of magnesium in the case of the alloy Pb2% Cd1% Bi with the same proportions of magnesium.

In addition, by comparing the quaternary systems Pb-Cd-Bi-Sn and Pb-Cd-Bi-Mg, it is found that the two types of alloys show a remarkable improvement in mechanical properties. Is that the combination of these elements by combining magnesium with tin and add the ternary system Pb-Bi-Cd could further increase the hardness?

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