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Effect of aging and step –quenched heat treatment on the Martensitic transformation of CuZnAl shape memory alloy

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Abstract:

There are many parameters affecting on the martensitic transformation of nonferrous alloys such as point defects, pores and heat treatment. In this work, the effect of aging after direct quenching treatment and step-quenched treatment on the Martensitic transformation of porous CuZnAL alloy are studied. X-ray diffraction analysis is based to identify phases presence in the microstructure of examined specimens. Optical microscopy and scanning electron microscopy examinations have also studied for prepared samples. It has been shown that aging and step-queched heat treatment lead to precipitate some intermetallic compounds in the Martensite matrix which make the lower percentage of shape memory effects for prepared alloy.

Key words: Martensitic transformation, aging, step-quenched , copper zinc aluminum, shape memory alloys.

تأثيرمعاملات الازمان و الإخماد المرحلي على التحول المارتنسايتي لسبيكة تستعيد شكلها من نحاس زنك المنيوم

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الخلاصة:

توجد العديد من العوامل المؤثرة على التحول المارتنسايتي في السبائك اللاحديدية مثل العيوب النقطية و المسامات والمعاملات الحرارية.في هذا العمل تم دراسة تاثير الازمان بعد الاخماد المباشر وكذلك تاثير معاملة الاخماد المرحلي على التحول المارتنسايتي لسبيكة من النحاس والزنك و الالمنيوم المسامية و المحضرة بتقنية ميتالورجيا المساحيق .اعتمد تحليل حيود الاشعة السينية للتعرف على الاطوار المتواجدة في بنية السبيكة المحضرة.تم فحص البنية المجهرية باستخدام المجهر الضوئي والمجهر الالكتروني الماسح.لقد اوضحت النتائج ان معاملة الازمان و معاملة الاخماد المرحلي تؤدي الى ترسيب طور شبه معدني (طور كاما) في اساس من المارتنسايت والذي يقلل بدوره من النسبة المئوية لتاثير استعادة الشكل.

الكلمات الدالة: التحول المارتنسايتي، الازمان،الاخماد المرحلي،نحاس زنك المنيوم،السبائك التي تستعيد شكلها.

(Wayman and Duerig, 1990). Shape memory alloys (SMAs) may sense thermal , mechanical , magnetic or electric stimulus and exhibit actuation or some predetermined , response which making it possible to tune some technical parameters such as shape , position , strain natural frequency damping and other static and dynamic characteristic of material systems in the response to the environmental changes. (Wei et al , 1998).The martensitic transformation is diffusion less that is no redistributions are permitted to occur during the transformation . It is a phase transition of first order involving changes in the crystal structure . (Ahlers , 2002) ,

Although martensitic transformation is still a subject of dispute , it seems that on the level of lattice dynamics almost first order martensitic transformation can fined a common origin which is driven dominantly by phonon entropy difference between parent phase and martensite . The parent phase has higher vibration entropy than that is in martensite phase .(Otsuka and Ren , 1999).

Martensite aging effect is an unclear phenomenon for over 60 years and has been found in many systems such as Au + Cd , Au+Cu+Zn , Cu+Zn +Al, Cu+Al+Ni and In –Ti (Otsuka and Ren, 1999. Martensite aging includes two time –dependent phenomena during aging . The first is called martensite stabilization whereas the Second called as rubber like (hyperelastic) behavior .(Otsuka and kakashita, 2002).

Martensite stabilization refers to appear the martensitic structure as more stable compared with parent (β) phase during aging where as rubber like behavior refer to the phenomenon that the martensite phase has a recoverable supperelastic deformation after deformation after aging.In copper base shape memory alloys ,both austenite(β) phase and martensite are susceptible to aging effects even at moderate temperatures (Wu,1990).When CuZnAl alloys is stabilized by post-quench aging and is them subjected to temperature range of 100- 350 ^{o}C , the change in transformation temperatures can be classified into three regions as shown in Fig.(10).(Wu, 1990). The first is described by a rapid drop of transformation .Temperature which are then recoverable during subsequent aging at temperature below 7^{o}C. The a mount of depression increase with aging temperature up to 300 °C and then decrease with further increases in the aging temperature. In the second stage, the transformation temperature increase moderately with time whereas in the third stage ,the hysteresis is widened significantly(Wu,1990).Several studies had been done for stabilization of martensite by aging and through using step- quenched technique. All of them applied two processes on the cast CuZnAl , CuAlNi and CuAl Be. In 1984 van Humbeek et al investigated stabilization of CuZnAl martensite through step-quenched heat treatment by the studying of reverse transformation temperature. Delaey et at , 1984 studied crystal structure and ordering through the stabilization of step-quenched CuZnAl martensite prepared by casting. Van Humbeek et al in 1985 presented interpretation of stabilization of step-quenched CuZnAl alloy using pistron annihilation whereas Graef et al ,1985 studied the changes in the microstructure during stabilization of CuZnAl martensite by step-quenching heat treatment using transmission electron microscope .(TEM).They showed that there is evidence for the presence of least pre-precipitation in the stabilized martensitic alloys. Thermal analysis data for Cu+25wt % Zn + 4 wt% Al ribbons stabilized by rapidly quenching process was presented by Scarsbrook and Stobbs ,1987. The training process for Cu+ 6.57 wt % Al + 20.18wt % Zn + 0.72 wt % Co alloy was presented by Mellor et al, 1990. (Pons and cesari , 1999) investigated interaction between γ-phase precipitation and martensite in CuZnAl alloy after flash heating using TEM.The changes in space lattice in Cu+24.98 wt % Zn + 4.43 wt % Al martensite during aging process were studied by (Cakmak et al ,2001).They used diffraction line profile obtained by X-ray diffractometer. Isothermal decomposition of martensite phase in CuZnAl alloys was studied by Asanovic etal 2006. They showed that the aging process at temperature below 400 ^{o}C involved the formation of bainite or α phase. As can be seen from literatures few works about aging and step quenched heat treatment had been done for CuZnAl alloys prepared by powder metallurgy. The aim of this work is to study the effect of aging and step quenched treatments on the martensitic transformation formed by direct quenching.

2.Experimental Procedure :

The porous Cu+21% Zn + 5.35 wt % Al shape memory alloy has been prepared by powder metallurgy technique by the same author (Abid Ali,2008) from elemental powders of Cu ,Zn and Al. In addition, martensitic transformation by direct quenching treatment has also been presented by (Abid Ali ,2008).The porous Cu+21wt % Zn + 5.35 Wt % specimens with (15 mm in diameter and 5 mm in hight) prepared with 11% porosity is heat treated with solution heat treatment upto 850 ˚C for one hour and followed by quenching in iced water (2-4 °C) and then aged in 200 ^{o}C and in 450 ^{o}C for 30 minutes with different cooling rate another sample is treated with step-quenched as follows:

1-heating the specimen upto 850 °C for one hour.
2-quenching in hot water 95^{o}C for 15 minutes and
3-quenching in iced water .

The heat treatment is done in Argon atmosphere with 1.5 bar / min rate in tube furnace as shown in Fig.(2).These equipments are in material engineering department ,metallurgical laboratory. Step-quenched heat treatment carried out in hot water with hot stage stirrer supplied with thermostat up to 250 ^{o}C. After aging in 200 ^{o}C and 450 ^{o}C and step-quenched treatment ,X-ray diffraction analysis are carried out for these specimen in S.C. Geology Survey and mining, Baghdad. The specimen was held at a fixed angle of θ=5 degrees relative to the incident X-ray been the detector was moved through 2θ= 10 to 90 degrees.The reference sample is direct quenched in iced water to compare with aged and stepped-quenched specimens with respect to X-ray diffraction (XRD) patterns. Another specimens are grinded silicon carbide paper with (80 ,400, 800 and 1200 grit and polished with fine (ϕ=0.025) alumina to prepare specimens for microstructure . examination with etching and without etching by light optical microscope (LOM) and with scanning electron microscope (SEM) which is done in advanced metallographic lab., Faculty of engineering ,Tehran university. The etching solution used to etch specimens is ($FeCl_3$ + HCl + H_2O) solution according to (Ricksecker ,1984).

3. Results and discussion ;

Fig. (3) shows the X-ray diffraction patterns for reference case which is direct quenched specimen and aged in 200 ^{o}C for 30 minutes in Fig.(4) respectively. It is shown that decomposition of orthorhombic martensite phase in to precipitates in the matrix of martensite . However , there is still martensite phase peeks. In Fig(5) , XRD pattern for aging in 450 ^{o}C after iced quenching treatment is shown that almost of martensite phase is decomposed in to $\alpha+\gamma$ phase and the intermetallic compound of γ is precipitated on the structure. Small amounts from martensite is also remained in the structure because the time of aging in 450 ^{o}C is low (30) minutes which does not allow to precipitate all γ phase. Fig. (6) Shows the XRD pattern for step-quenched specimen. It is clear that some of intermetallic monoclinic compound is found in the structure in addition to presence of orthorhombic marten site phase.

Light optical microscope(LOM) images for aged and step-quenched specimens are shown in Fig.(7) and Fig.(8) respectively.

Precipitates of γ phase unfortunately do no appear in LOM microstructures. These precipitate required transmutation electron microscope to reveal its crystalline structure and morphology. Scanning electron microscopy images of aged in 200^{o}C specimen and step- quenched heat treated specimen are shown in Fig.(9) and Fig.(10) respectively. It is clear that monoclinic γ

phase is precipitated along the grain boundaries as black spots near the grain boundaries as shown in Fig.(8). It is also clear as can be seen from Fig.(9) that γ phase is precipitated in the microstructure of step-quenched specimen.

4. Conclusions.

From the X- ray diffraction patterns of aged specimens and step-quenched specimens in addition to SEM images. It is concluded that

1- Post-quenching aging heat treatment of prepared Cu Zn Al alloy at 200 ˚C or at 450 ˚C lead to precipitate γ (monoclinic) phase in the microstructure of alloy .

2- Step-quenched in 95 ˚C causes precipitate of γ (monoclinic) phase in the matrix of martensite phase .

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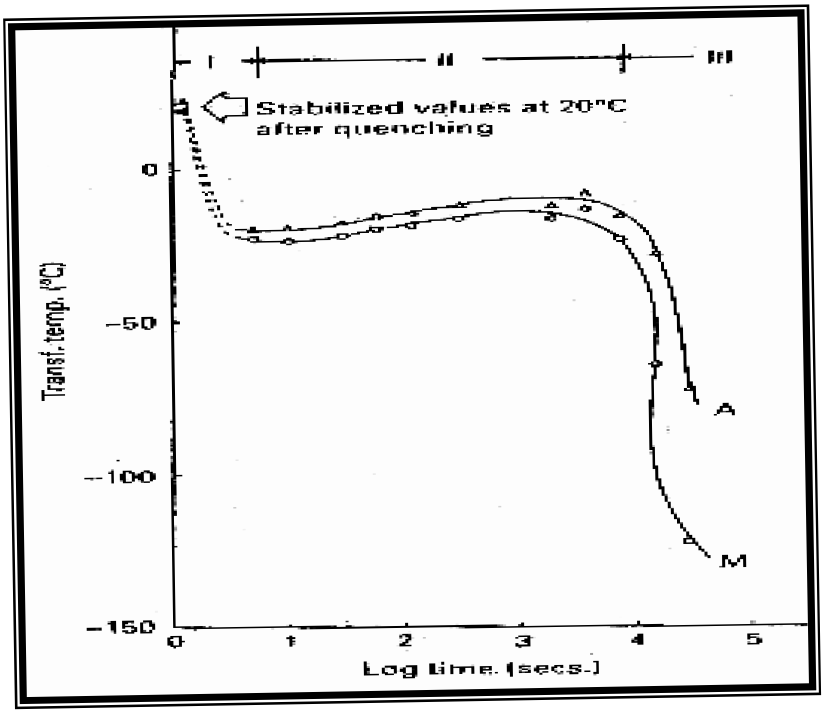

Fig. (1) Changes in M and A temperatures of cu-26.67Zn-4Al alloy during aging at 200 C° .(adapted from Wu,1990) .

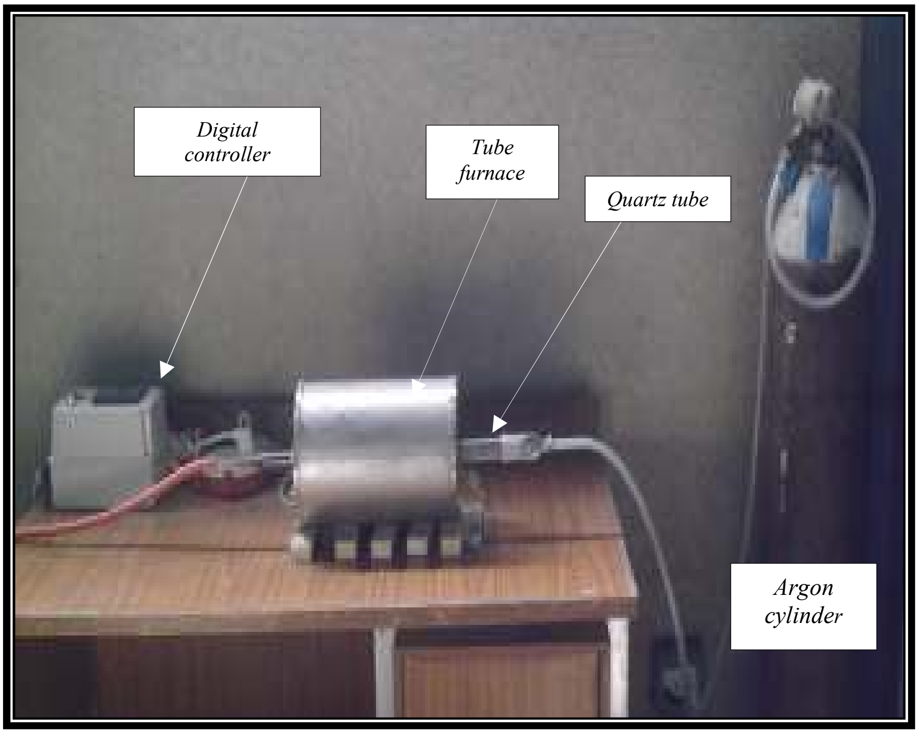

Fig. (2) Tube furnace used in this study.

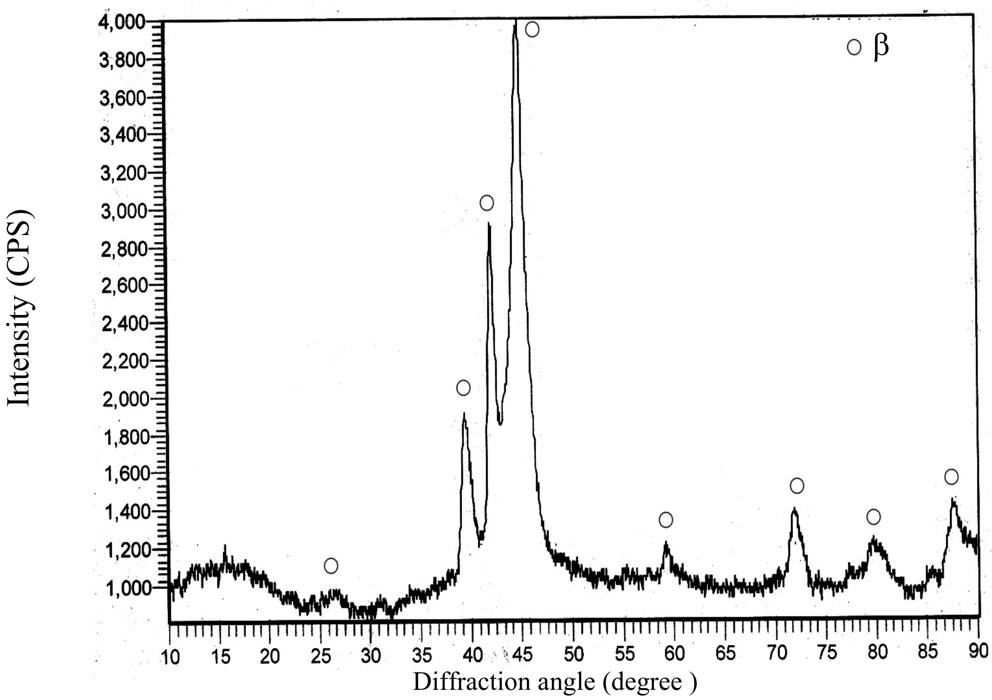

Fig.(3) XRD pattern for direct quenched in iced water.

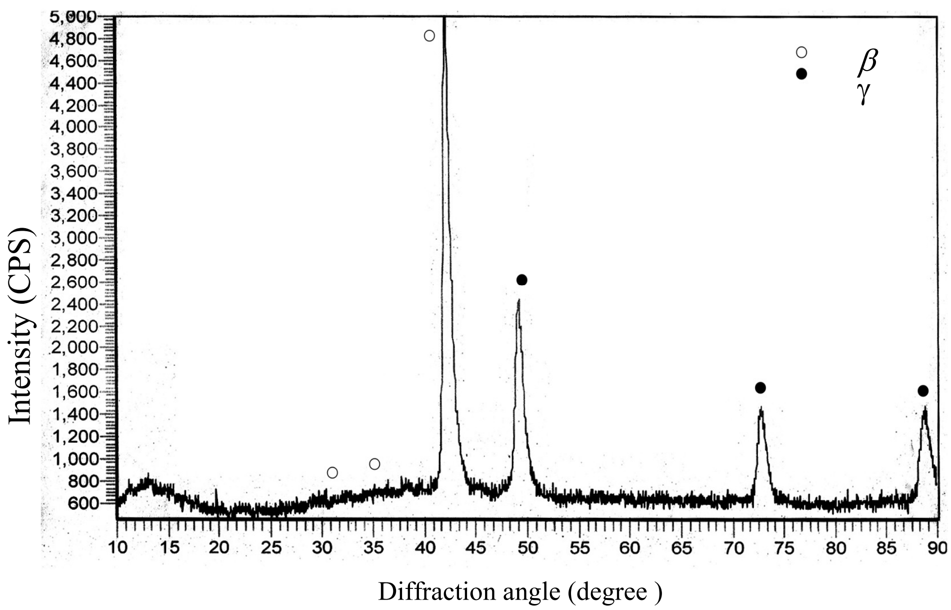

Fig.(4)XRD pattern for aging at 200 ^{o}C for 30 min and iced quenched .

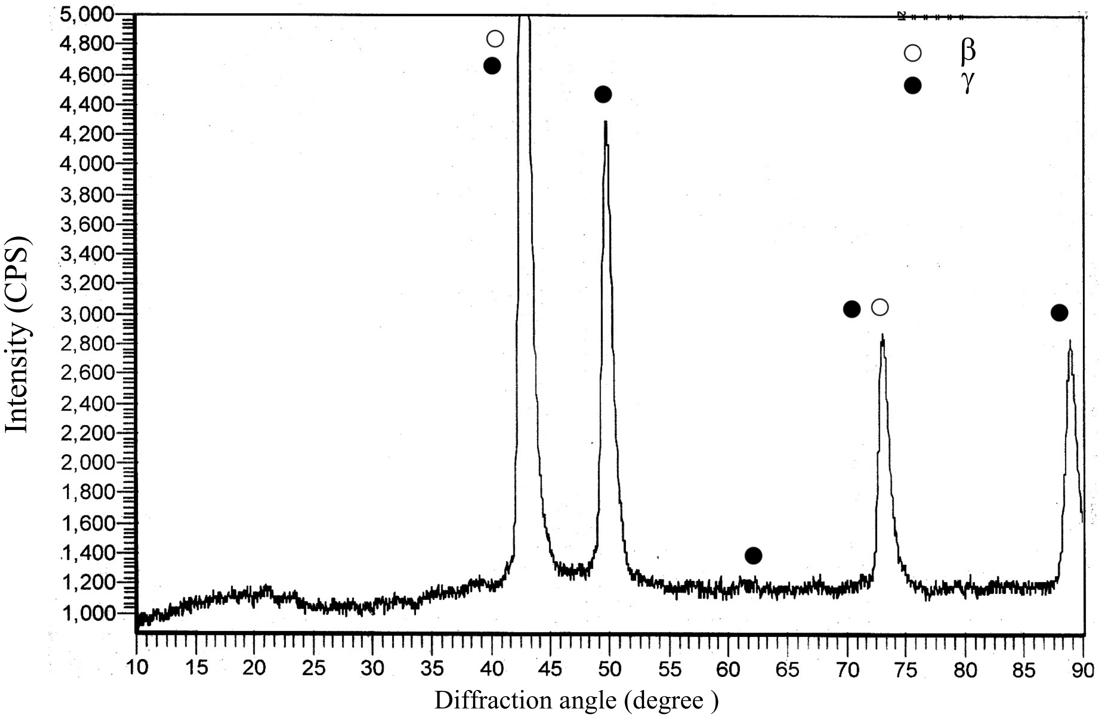

Fig.(5)XRD pattern for aging at 450 ^oC for 30 min and air cooled .

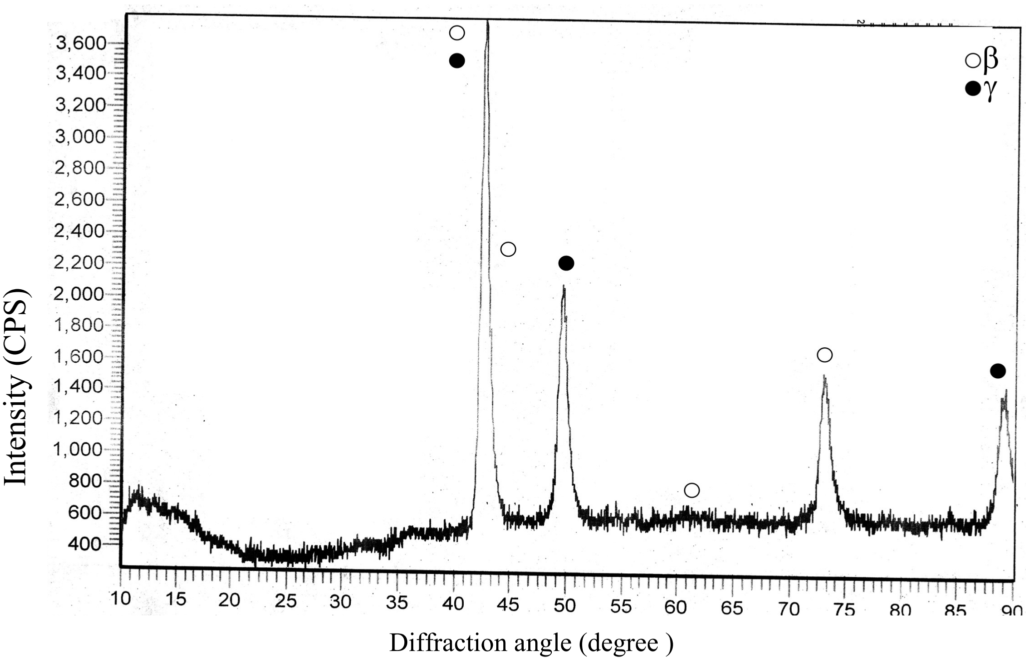

Fig.(6)XRD pattern step quenched in 95 ^oC for 15 minutes and iced quenched .

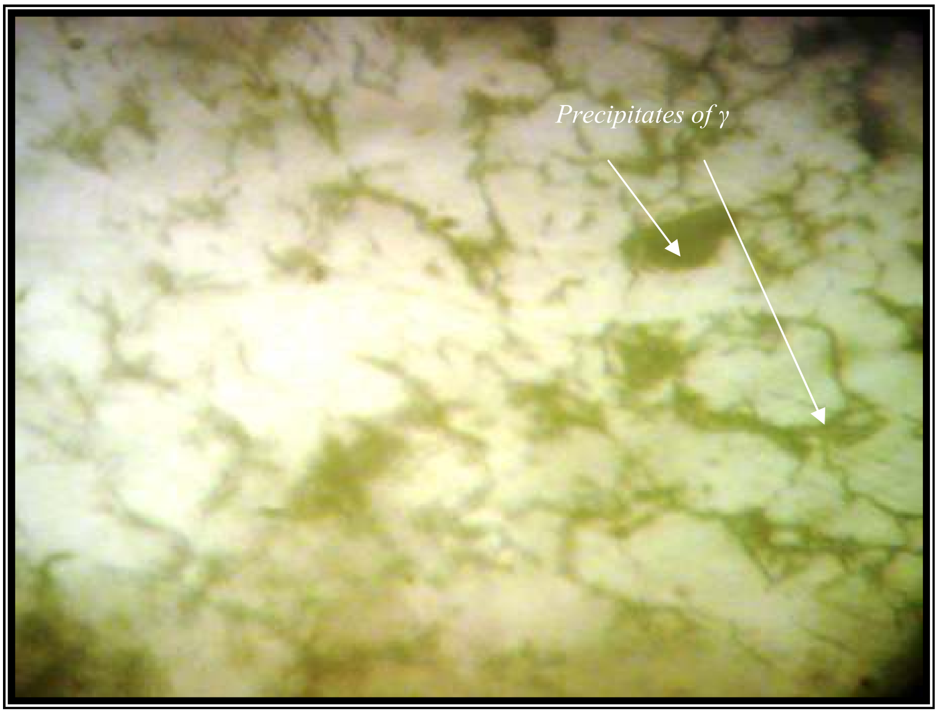

Fig.(7) Light optical microscope image for aged specimen.(X1000)

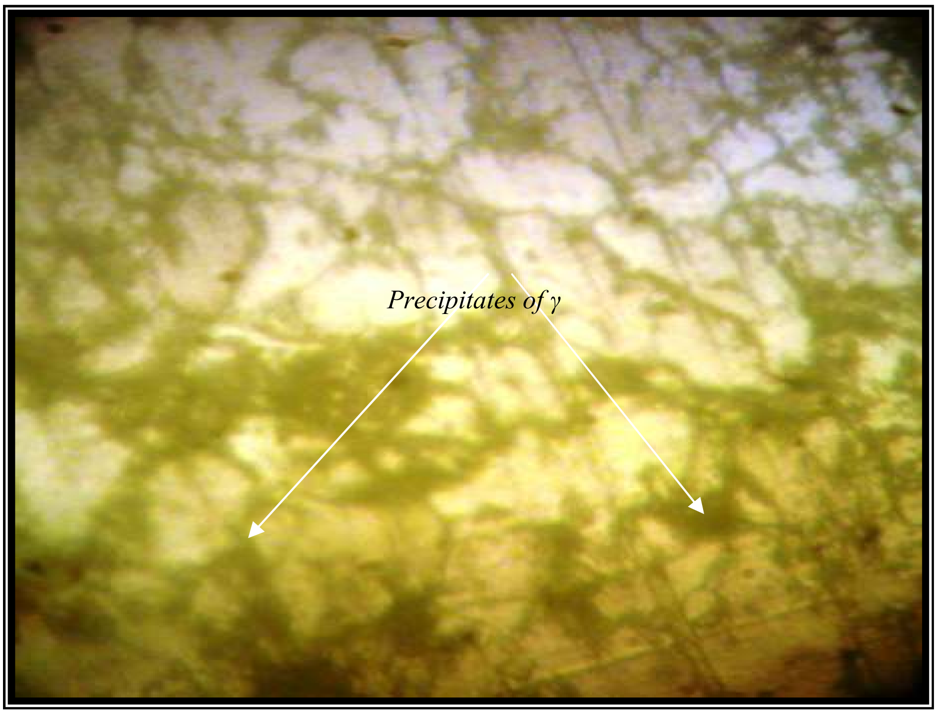

Fig.(8) *) Light optical microscope image for step quenched specimen.(X1000)*

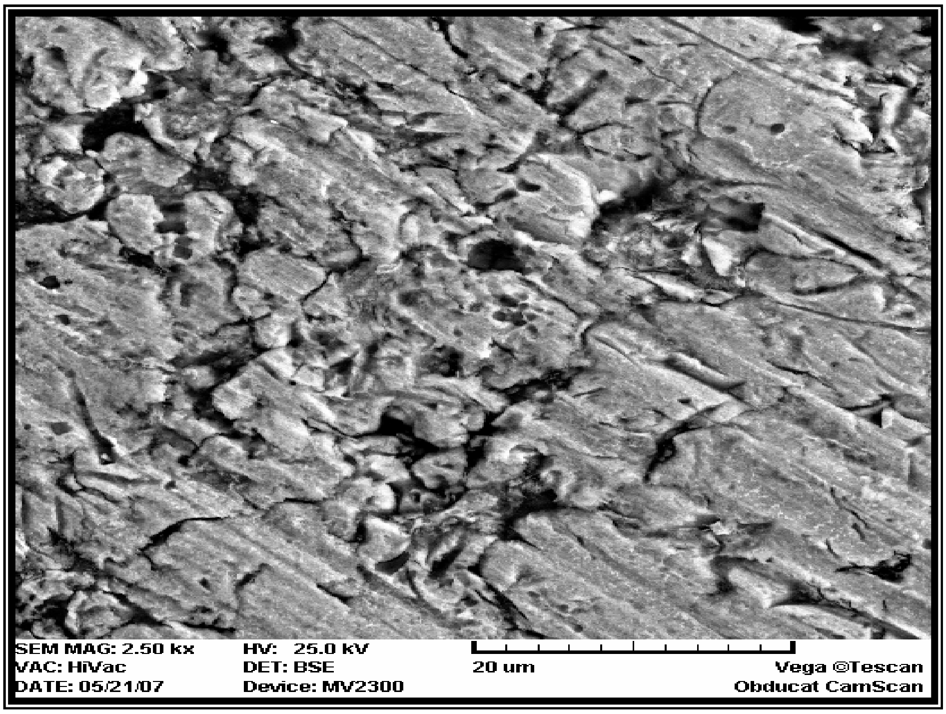

Fig.(9) SEM image for aged specimen.
(etched with FeCl3+HCl+H2O)solution.

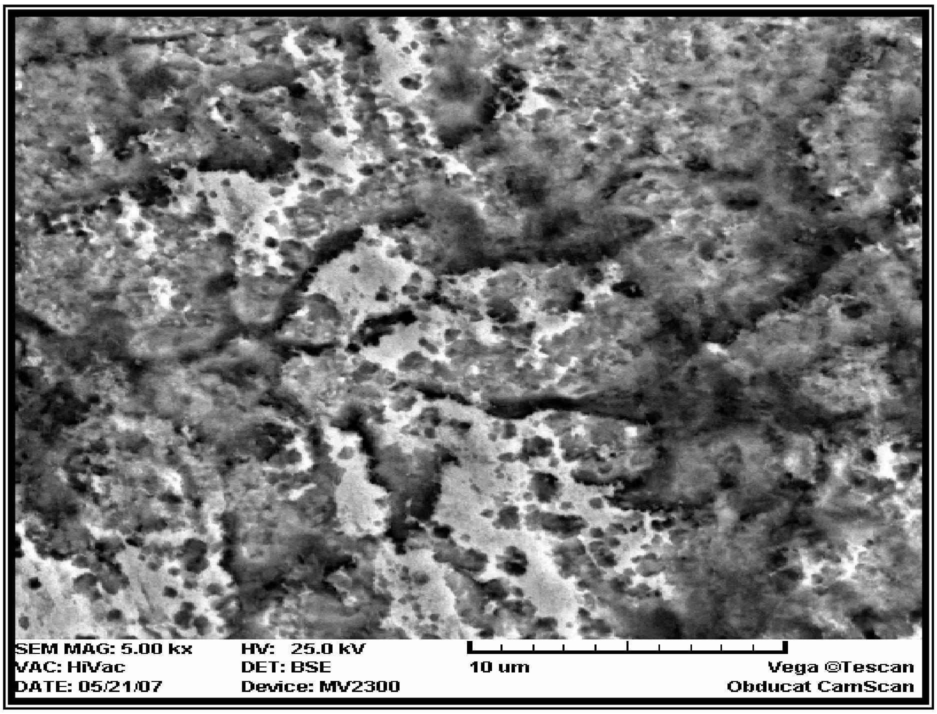

Fig.(10) SEM image for step quenched specimen
(etched with FeCl3+HCl+H2O)solution.