

# Effect of prior cold work on age hardening of Cu-4Ti-1Cd alloy

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This research is part of a project whose scope was to develop high strength ternary alloys based on Cu-Ti system with the primary aim of substituting them for toxic and expensive Cu-Be alloys. In this pursuit, age hardening behaviour of Cu-4Ti-1Cd alloy has already been investigated and the present paper reports the investigations on the influence of prior cold work by rolling of 50, 75 and 90% on the age hardening of a Cu-4Ti-1Cd alloy using hardness and tensile tests and optical as well as transmission electron microscopy. As a result of cold work followed by aging, hardness of the alloy increased from 237 Hv in solution treated condition to 425 Hv on 90% cold work and peak aging. Similarly, yield and tensile strengths of the alloy reached maxima of 1037 and 1252 MPa respectively on 90% deformation and peak aging. The microstructure of the deformed alloy exhibited elongated grains and deformation bands. The maximum strength on peak aging was obtained due to precipitation of ordered, metastable and coherent  $\beta^1$ ,  $\text{Cu}_4\text{Ti}$  phase in addition to high dislocation density and deformation twins. Both hardness and strength of the alloy decreased on overaging due to the formation of incoherent and equilibrium  $\beta$ ,  $\text{Cu}_3\text{Ti}$  phase. However, the morphology of the discontinuous precipitation was changed to globular shape due to large deformations and overaging.

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## 1. Introduction

Cu-Ti alloys have been developed with the aim of substituting them for toxic and expensive Cu-Be alloys. Binary Cu-Ti alloys are known to gain high strength through age hardening [1, 2]. Earlier studies [3] have shown that Cu-Ti alloys are precipitation strengthened on aging by the spinodal decomposition mechanism involving clustering and ordering in the initial stages. An extensive work on precipitation mechanism in Cu-Ti alloys [3–6] indicates that an intermetallic phase  $\beta^1$  ( $\text{Cu}_4\text{Ti}$ ) would contribute to strengthening during aging. Prolonged aging of Cu-Ti alloys would lead to cellular precipitation along the grain boundaries of the matrix and the forma-

tion of an equilibrium precipitate,  $\beta$  ( $\text{Cu}_3\text{Ti}$ ) [1, 4, 7]. Numerous research on Cu-Ti alloys found that these alloys have potential as substitute for Cu-Be alloys because of their comparable strength and electrical conductivity [5, 6, 8].

Nagarjuna *et al.* [8] studied the structure-property correlations of Cu-Ti alloys in various conditions, viz. solution treatment, solution treatment & aging and solution treatment, cold work & aging. It was reported that significant microstructural changes did not occur due to prior cold work but the tensile properties of Cu-Ti alloys improved greatly and matched with those of Cu-Be-Co alloys.

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The aim of the present work was to investigate the effects of alloying additions to the binary Cu-Ti alloys in order to further enhance their strength. In this pursuit, our recent studies on the addition of cadmium to Cu-4Ti alloy [9] revealed that the tensile properties of the alloy increased considerably in solution treated condition due to substitutional solid solution strengthening by Cd and delay in peak aging compared to binary Cu-Ti alloys. The tensile properties of ternary Cu-4Ti-1Cd alloy were also comparable to those of Cu-2Be-0.5Co alloy [10] in undeformed condition. The strengthening mechanism and microstructure of Cu-4Ti-1Cd alloy were similar to that of binary Cu-4Ti alloy. Cold deformation was reported to improve the strength of metals and alloys considerably [5, 6, 8, 11, 12]. The effects of cold deformation on age hardening of Cu-4Ti-1Cd alloy have not been reported so far and therefore, we have attempted to fill this gap by investigating the structure and properties of

solution treated Cu-4Ti-1Cd alloy subjected to cold work followed by ageing. The present paper reports results on hardness, tensile properties, microstructural features and age hardening mechanism of Cu-4Ti-1Cd alloy in solution treated, cold worked and aged condition.

## 2. Experimental procedure

Cu-4Ti-1Cd alloy was prepared by melting pure metals of Cu, Ti and Cd in a STOKES Vacuum Induction Melting furnace. The ingot was homogenized at 800°C for 24 h and analysed for Ti and Cd. The analysis reported Ti and Cd as 3.96 and 0.99 wt.% respectively. The ingot was subsequently hot forged and rolled at 850°C into rods of 12 mm  $\phi$  and sheets of 10 mm thickness. Small samples of the hot rolled alloy were solutionized at 860°C for 2 h followed by rapid quenching in water. The solutionised samples were subjected to different amounts of cold

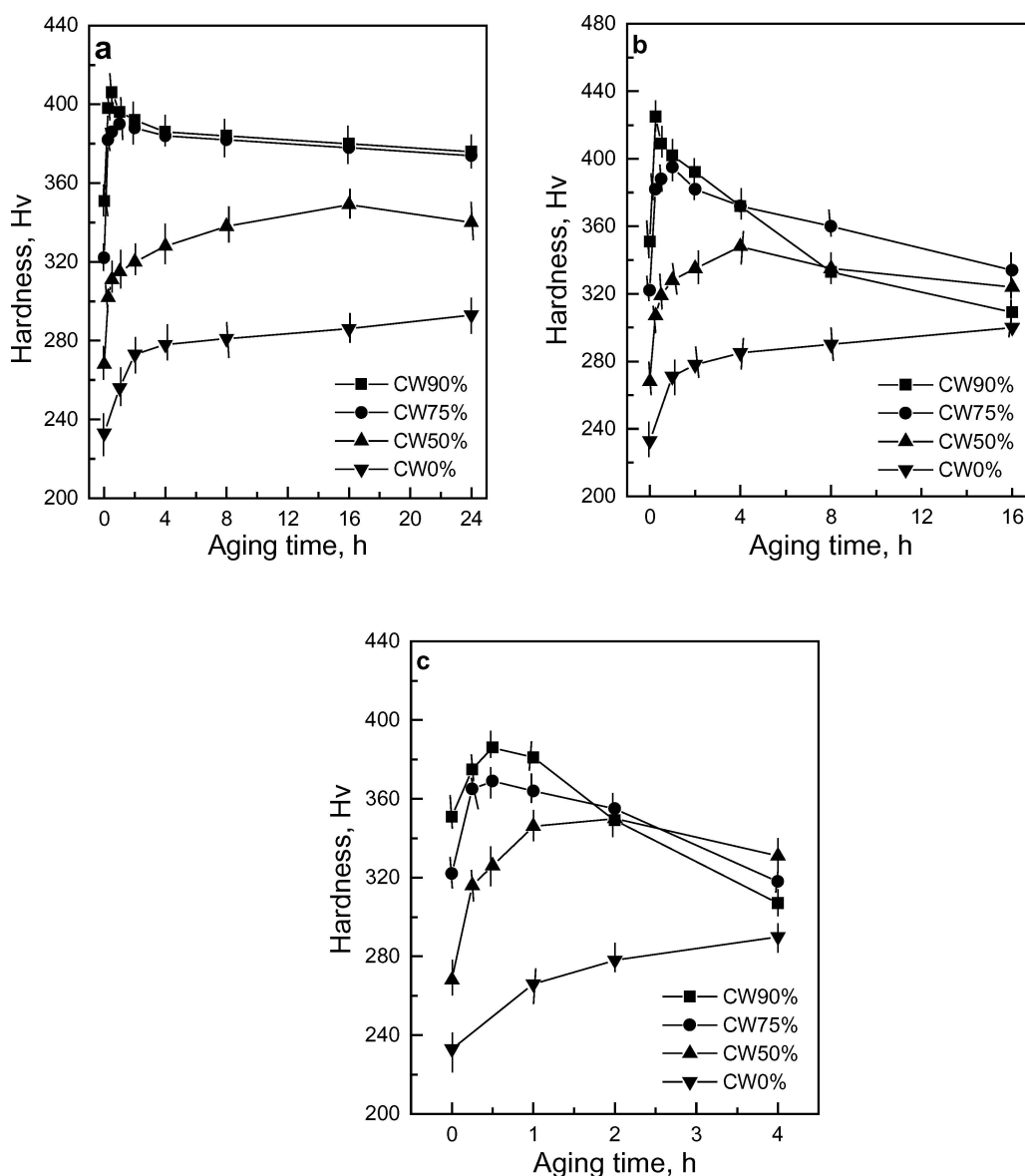


Figure 1 Effect of prior cold work on age hardening of Cu-4Ti-1Cd alloy at (a) 400°C (b) 450°C and (c) 500°C.

deformation, viz. 50, 75 and 90% by rolling at room temperature. Age hardening studies were conducted on samples with the above deformations at 400, 450 and 500°C to generate data on peak aging time and temperature for each deformation. Vickers hardness (at 10 kg load) of these samples was measured at frequent intervals at all the temperatures studied. Five readings were taken for each hardness measurement and the average is reported here.

Flat tensile samples of the alloy with the said deformations were peak aged at the temperature at which maximum hardness was observed (450°C) and tested for tensile properties at room temperature at a nominal strain rate of  $10^{-3} \text{ s}^{-1}$  using INSTRON tensile testing machine. Optical metallographic analysis was carried out on mechanically polished and etched samples with the etchant being a mixture of 10 g  $\text{K}_2\text{Cr}_2\text{O}_7$ , 5 ml  $\text{H}_2\text{SO}_4$ , few drops of HCl and 95 ml distilled water. Thin slices were cut from the bulk of the deformed alloys using low speed ISOMET cutting machine and then mechanically polished to 50–60  $\mu\text{m}$  thick slices. Three mm discs were punched out from these slices. Electropolishing of these discs was done using twin jet electropolisher in a solution containing 30 vol%  $\text{HNO}_3$  and 70 vol% methanol at  $-45^\circ\text{C}$  and 20 V. TEM analysis was carried out on the samples in different conditions of the alloy using 160 kV in a JEOL 200CX Transmission Electron Microscope.

### 3. Results and discussion

#### 3.1. Hardness

The effect of aging time at 400, 450 and 500°C on the hardness of Cu-4Ti-1Cd alloy deformed to 50, 75 and 90% reduction in thickness is shown in Fig. 1, which also includes the hardness of undeformed alloy [9]. The alloy with 50% deformation attained a peak hardness of 349 Hv after aging for 16 h at 400°C, 75% deformed alloy attained a peak hardness of 385 Hv in 1 h and 90% deformed alloy, 410 Hv in 30 min, while the undeformed alloy did not show peak hardness even after aging for 24 h at 400°C (Fig. 1a). At the aging temperature of 450°C (Fig. 1b), the 50% deformed alloy peak-aged in 4 h (350 Hv) while the 75% deformed alloy in 1 h (395 Hv) and the 90% deformed alloy in just 15 min (425 Hv). Overaging at this temperature was very fast in 90% deformed alloy as compared to other deformations. At 500°C (Fig. 1c), the 50% deformed alloy attained a peak hardness of 348 Hv in 2 h while that with 75 and 90% deformations attained peak hardness of 369 Hv in 1 h and 386 Hv in 15 min respectively. The peak hardness of 75 and 90% deformed alloys was lower at 500°C than that at 450°C.

Variation of hardness with % cold work of the solution treated and cold worked Cu-4Ti-1Cd alloy peak aged at 400, 450 and 500°C is shown in Fig. 2. In the solution treated and undeformed condition, the hardness of the alloy increased from 238 Hv to 260 Hv on 50% defor-

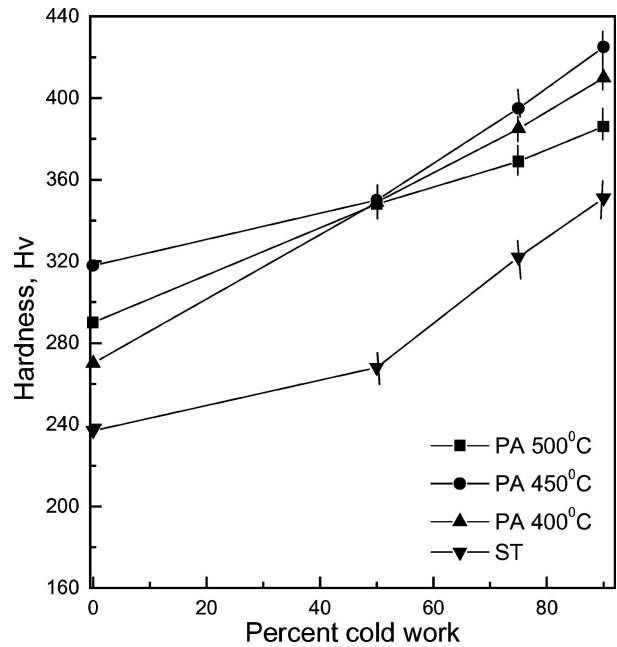


Figure 2 Effect of prior cold work on hardness of Cu-4Ti-1Cd alloy in solution treated and peak aged conditions.

mation but it increased rapidly on further cold working to reach a maximum of 351 Hv on 90% cold work. The peak hardness was maximum for all the deformations of the alloy when aged at 450°C. The highest hardness for Cu-4Ti-1Cd alloy recorded (425 Hv) for 90% cold work followed by aging at 450°C for 15 min (Fig. 1b) was almost the same as that reported for the binary Cu-4.5Ti alloy by Nagarjuna *et al.* [8]. The peak aging time was reduced to 15 min for the alloy with 90% deformation against 40 h in the undeformed condition. It is worth to be noted here that the hardness of Cu-4Ti-1Cd alloy is much better than those of other Cu based precipitation-hardening alloys such as Cu-Fe-Cr or Cu-Cr-Fe-X alloys reported by Fernee *et al.* [13–15]. While Cu-4Ti-1Cd alloy exhibits a maximum hardness of 425 Hv, the Cu-Fe-Cr alloys showed only 171 Hv in 90% CW + aged condition.

The present investigation shows that there is a considerable increase in hardness after cold work in solution treated condition of Cu-4Ti-1Cd alloy. The maximum increase in hardness is limited to about 50% with 90% deformation. The increased dislocation density as a result of cold work caused the increase in hardness, which is in agreement with the literature [12]. The contribution of prior cold work in increasing the hardness of the alloy is not considerable; since the alloy exhibited high hardness in the solution treated condition itself (238 Hv) due to the solid solution strengthening effect of cadmium and modulated structure containing fine precipitation of  $\beta^1 \text{Cu}_4\text{Ti}$  phase [9]. On aging the cold worked alloy, hardness increased by 80% reaching a peak value of 425 Hv with 90% deformation and peak aging at 450°C. When the solution treated alloy is cold worked prior to aging, dislocation density increases which enhances the strength due to work hardening. Further, on aging the deformed

TABLE I Comparison of mechanical properties of Cu-4Ti-1Cd with those reported for Cu-4.5Ti [8] and Cu-2Be-0.5Co [10] alloys

Property	Solution treated		Cold work, % + Peak aged at 450°C		Cu-4.5Ti	Cu-2Be-0.5Co
	0%	0% (40 h)	50% (4 h)	90% (15 min)	ST + 90%CW + PA	ST + full hard + aged
YS, (MPa)	528	751	881	1037	1280	1140–1415
UTS, (MPa)	754	894	1084	1252	1380	1310–1480
%El	27	10	7.6	1.2	2	1–4
Hardness, (Hv)	238	318	350	425	425	385

ST: Solution Treated; CW: Cold worked; PA: Peak aged.

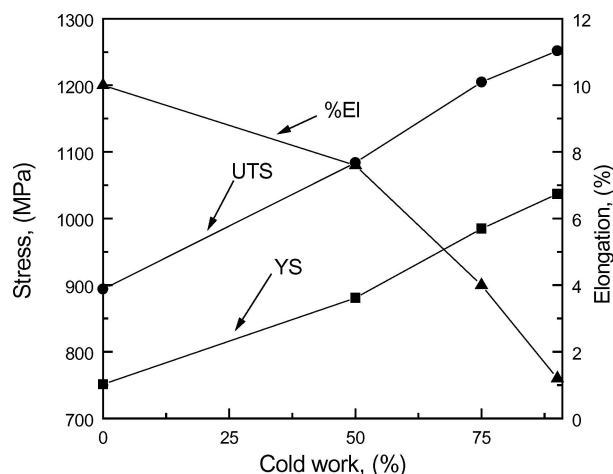


Figure 3 Effect of prior cold work on yield strength, ultimate tensile strength and elongation of Cu-4Ti-1Cd alloy peak aged at 450°C.

alloy, a fine dispersion of metastable precipitate, i.e.  $\beta^1$  Cu<sub>4</sub>Ti with increased volume fraction forms. This is reflected in TEM images of deformed alloy in peak-aged

condition (Figs. 6 and 7). Cold deformation prior to aging has also resulted in considerably reduced peak aging times as compared to the undeformed alloy [9]. The highest hardness value obtained for this alloy is comparable with those of binary Cu-4.5Ti [6, 8] and Cu-2Be-0.5Co alloys [10] (Table I). However, the peak aging temperature of cold worked ternary alloy is higher than that for the corresponding binary alloy, which is attributed to the influence of cadmium on the aging kinetics.

### 3.2. Tensile properties

The effect of prior cold work on yield strength (YS) and ultimate tensile strength (UTS) of Cu-4Ti-1Cd alloy peak aged at 450°C is shown in Fig. 3. Both YS and UTS increased with the amount of cold work and attained maximum values at 90% deformation. Hardness and tensile properties of the alloy in different conditions are compared with those of binary Cu-4.5Ti and Cu-2Be-0.5Co alloys in Table I. YS of the alloy increased from 528 MPa

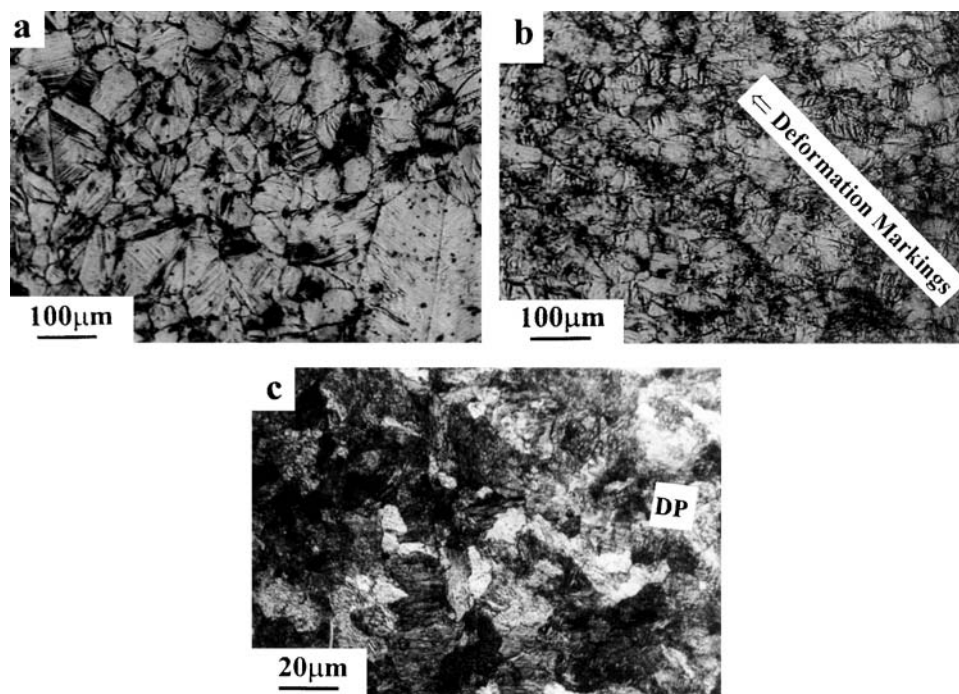


Figure 4 Optical micrographs of solution treated and 50% cold worked Cu-4Ti-1Cd alloy. (a) Peak aged at 450°C/34 h (b) overaged at 450°C/16 h and (c) overaged at 500°C/32 h.

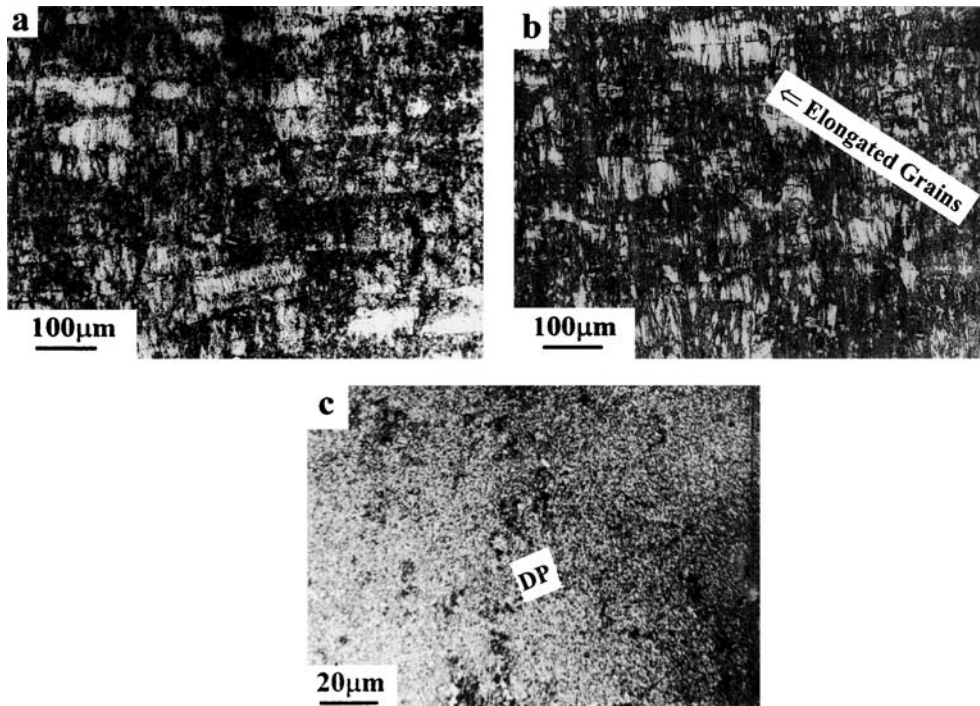


Figure 5 Optical micrographs of solution treated and 90% cold worked Cu-4Ti-1Cd alloy. (a) Peak aged at 450°C/15 min. (b) overaged at 450°C/16 h and (c) overaged at 500°C/32 h.

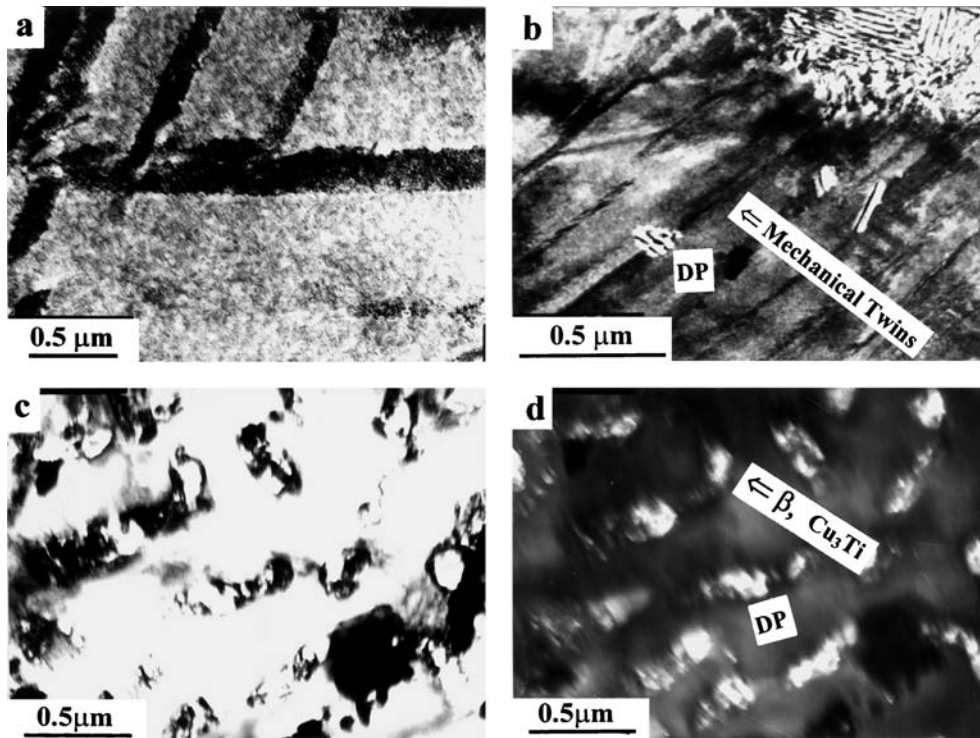


Figure 6 TEM images of solution treated and 50% coldworked Cu-4Ti-1Cd alloy. (a) Peak aged at 450°C for 4 h, (b) Overaged at 450°C for 16 h, (c) BF and (d) DF of overaged alloy at 500°C for 32 h.

in solution treated condition to 751 MPa on peak aging at 450°C. It further increased on prior cold work and aging to 881 MPa with 50% cold work and a maximum of 1037 MPa with 90% deformation (Fig. 3). Similarly, the UTS, 754 MPa in solution treated condition reached

to 894 MPa on peak aging at 450°C which was further augmented on cold work and aging to 1084 MPa with 50% cold work and 1252 MPa with 90% deformation (Fig. 3). However, the ductility (% elongation) decreased drastically to 1.2 with 90% cold work and peak aging

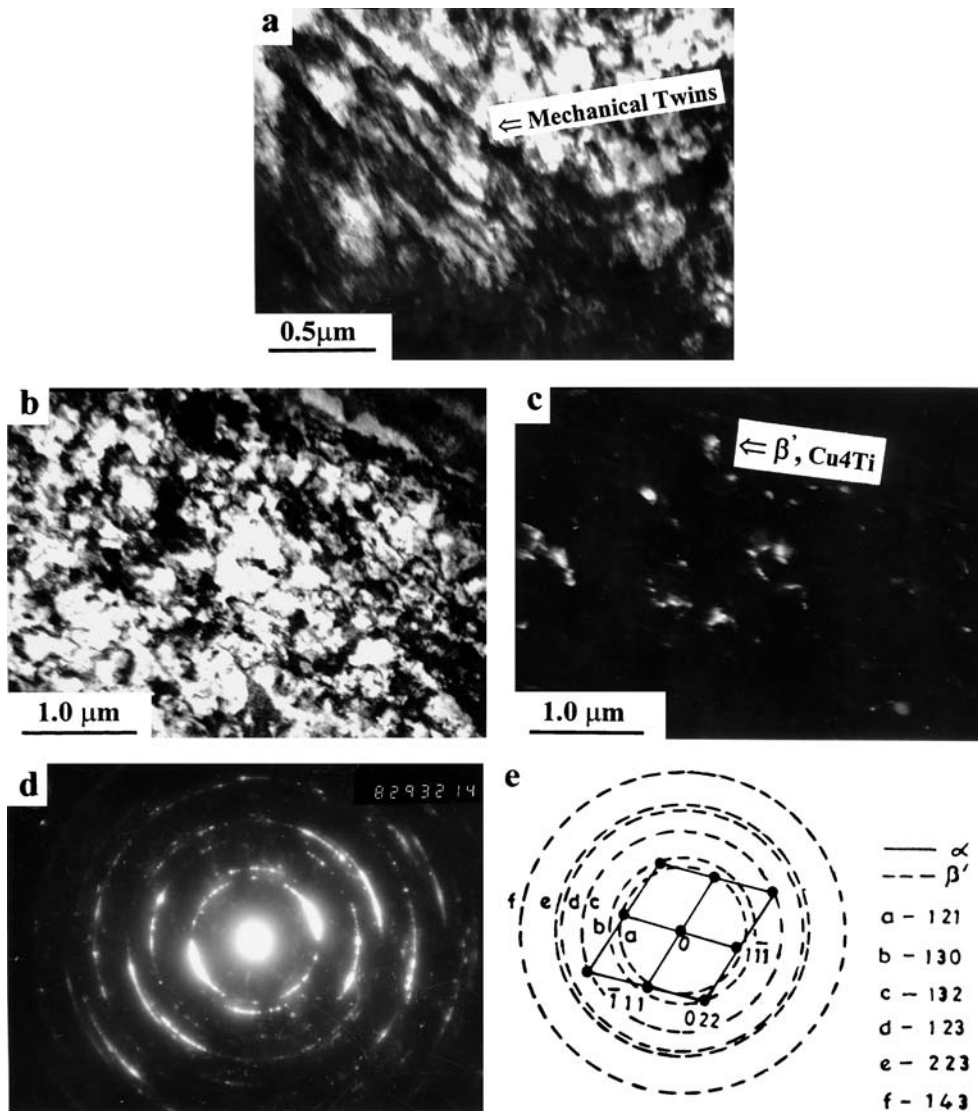


Figure 7 TEM images of solution treated and 90% coldworked Cu-4Ti-1Cd alloy peak aged at 450°C for 15 min. (a) BF of the matrix (b) BF (c) DF (d) SAD and (e) Schematic of SAD..

compared to 27% in solution treated condition and 10% in undeformed and peak-aged condition.

Both YS and UTS followed the trend of hardness exhibiting an increase on aging of the deformed alloy. The compositional modulations and presence of fine precipitates of  $\beta^1$  Cu<sub>4</sub>Ti resulted in higher YS and UTS in solution treated condition of the alloy [9]. The extent of increase in tensile properties (YS as well as UTS) of deformed alloy is nearly 17–20% in 50% deformed and peak aged alloy over the peak aged condition of undeformed alloy, while it is nearly 40% increase after 90% deformation and peak aging. Still, the YS and UTS values of Cu-4Ti-1Cd alloy are lower than those of binary Cu-Ti alloys. However, the strength properties of Cu-4Ti-1Cd alloy are certainly far better than those of other Cu based precipitation hardening alloys such as Cu-Fe-Cr or Cu-Cr-Fe-X alloys reported by Fernee *et al.* [13–15]. The YS of Cu-4Ti-1Cd alloy (1037 MPa) is greater by 122% while UTS

(1252 MPa), by 133% of Cu-Fe-Cr alloys (YS: 467 MPa & TS: 537 MPa) [14] in the similar condition, i.e. 90% CW + aged.

The precipitation of fine metastable and coherent phase  $\beta^1$  Cu<sub>4</sub>Ti with bct structure has played significant role in improving the tensile strength in 50% deformed and peak-aged alloy. In addition to the mechanical twins, dislocation cells formed due to cold work also participate in improving the properties significantly. Since increase in YS and UTS on 50% deformation and peak aging is only 15–20% over the peak aged alloy in undeformed condition, the precipitation hardening mechanism is predominant, similar to undeformed alloy, than the work hardening at lower deformations. With 90% deformation, the grains are severely deformed and dislocation density increased considerably. Due to the cell structure of dislocations and mechanical twins in the 90% deformed alloy, YS and UTS increased in the range of 35–40% over the undeformed

alloy on peak aging, indicating the major role of cold work in improving the tensile properties. The higher deformation (90%) enhanced the rate of formation of precipitate  $\beta^1$   $\text{Cu}_4\text{Ti}$  on aging by reducing the peak aging time.

### 3.3. Optical microscopy

The optical microstructure of undeformed Cu-4Ti-1Cd alloy in different conditions reported by us earlier [9] is briefly reviewed here for comparison. In the solution treated condition (860°C/2 h/WQ), the alloy essentially consisted of equiaxed grains and annealing twins. Peak aging at 450°C for 40 h did not show any change in the microstructure. However, overaging at 450°C for 80 h or at 500°C for 32 h resulted in discontinuous precipitation at the grain boundaries of the matrix [9].

In the present study, the microstructural changes in Cu-4Ti-1Cd alloy with prior cold work (after solution treatment) and aging are shown in Figs. 4 and 5. A single phase structure with slightly elongated grains in 50% deformed alloy peak aged at 450°C and severely elongated grains in 90% deformed alloy also peak aged at 450°C are seen in Fig. 4a and 5a respectively. The onset of discontinuous precipitation (DP) at a few grain boundaries of the matrix can be observed in 50% deformed alloy (Fig. 4b) as well as in 90% deformed alloy (Fig. 5b) when overaged at 450°C for 16 h. Considerable amount of matrix is covered in individual grains by discontinuous precipitation on 50% deformation (Fig. 4c) and spheroidisation of discontinuous precipitation on 90% deformation (Fig. 5c) on overaging at 500°C for 32 h. Fine deformation markings can be seen in peak aged as well as overaged conditions of the 50% deformed alloy (Figs. 4a and b) whereas extremely fine deformation markings persisted

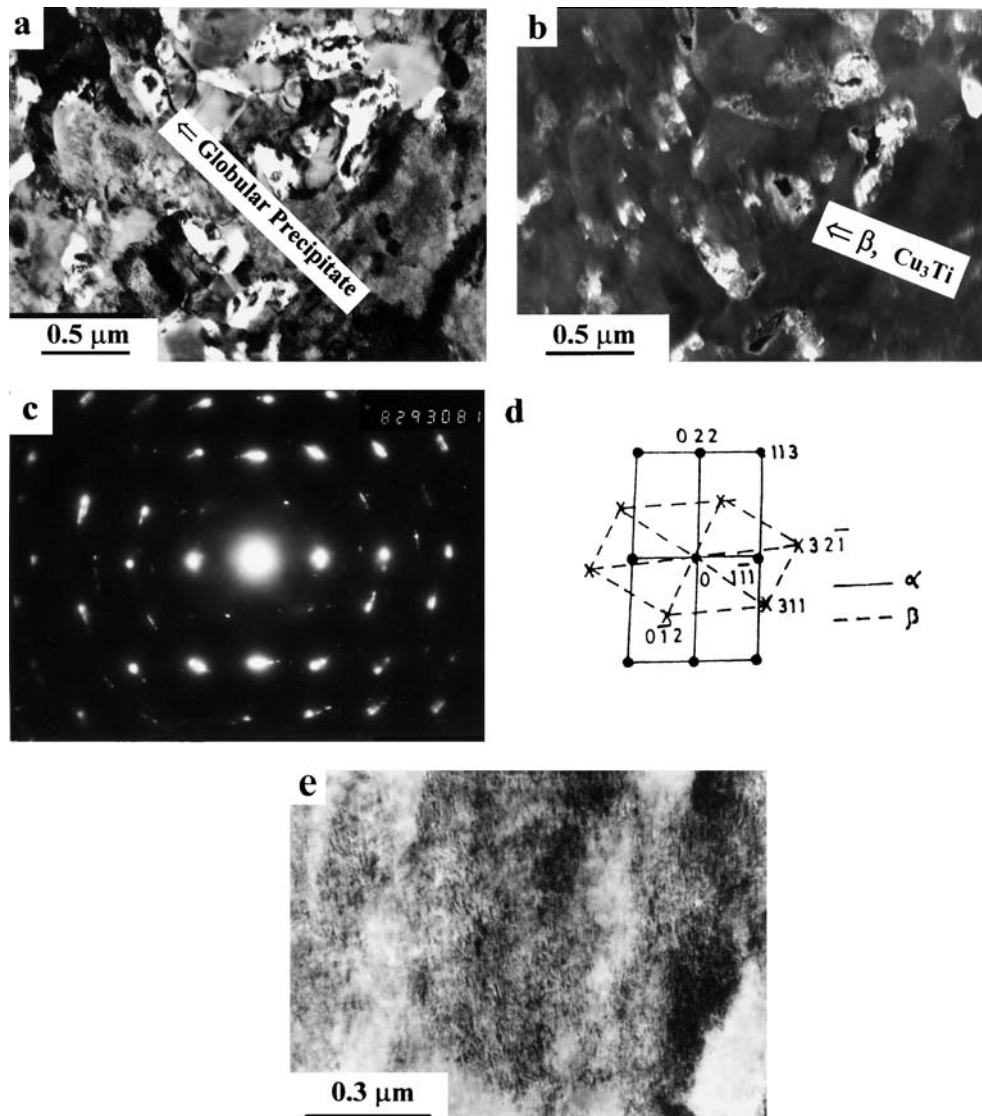


Figure 8 TEM images of solution treated and 90% coldworked Cu-4Ti-1Cd alloy overaged at 450°C for 16 h (a) BF and (b) DF of discontinuous precipitation (c) SAD and (d) Schematic of SAD and (e) BF of the matrix.

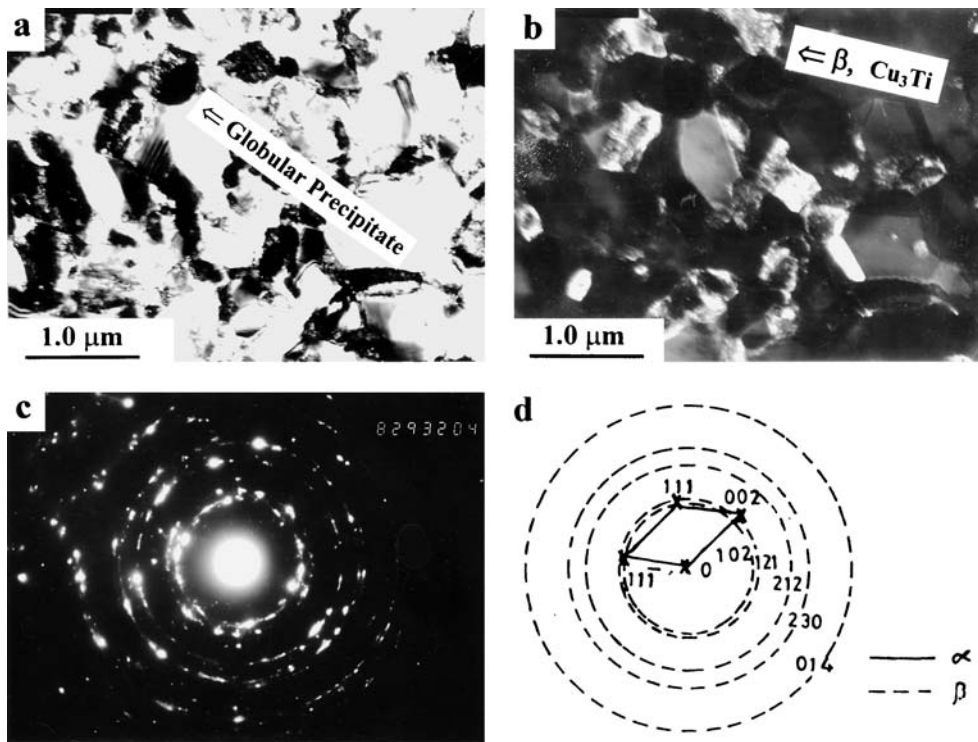


Figure 9 TEM images of solution treated and 90% coldworked Cu-4Ti-1Cd alloy overaged at 500°C for 32 h. (a) BF (b) DF (c) SAD and (d) Schematic of SAD of discontinuous precipitation.

in the severely deformed structures even on peak aging or overaging at 450°C (Figs. 5a and b). The observed microstructure in the present study is different from that of the undeformed alloy [9] and the effects of cold work, i.e. presence of elongated grains and fine deformation markings are clearly brought out in the microstructures (Figs. 4 and 5). It is however, similar to that reported in the binary Cu-Ti alloys [5, 6, 8]. As a result of cold rolling, the grains get deformed and appear as elongated domains in the direction of rolling [12]. The extent of elongation of the grains indicates the amount of cold work that the alloy has been subjected to, by cold rolling. Since the time and temperature of aging are not sufficient for recovery and recrystallisation to occur, the elongated grains and deformation markings persist in the microstructure in Figs. 4a, 4b, 5a and 5b. In the case of the overaged alloy at 500°C for 32 h, some recrystallisation appears to have taken place and the deformed microstructure has been replaced by the spheroidised microstructure showing discontinuous precipitation (DP).

### 3.4. Transmission electron microscopy

In the undeformed condition, Cu-4Ti-1Cd alloy exhibited compositional modulations and very fine precipitate of  $\beta^1$  Cu<sub>4</sub>Ti in the solution treated condition [9]. The modulated structure persisted on peak aging with increased quantity of metastable precipitate  $\beta^1$  Cu<sub>4</sub>Ti. The equilibrium precipitate  $\beta$ , Cu<sub>3</sub>Ti formed in the matrix on overaging. Discontinuous precipitation with lamellar morphology was

also noticed at the grain boundaries of the matrix when overaged either at 450 or 500°C.

In the present investigation, transmission electron microscopy of cold worked and aged Cu-4Ti-1Cd alloy with different deformations (50 & 90%) and aging times at 450 and 500°C was carried out to characterize and correlate with mechanical properties. Transmission Electron Micrographs (TEMs) of 50% cold worked and peak aged Cu-4Ti-1Cd alloy are shown in Fig. 6. Fig. 6a exhibited large number of twins with incoherent boundaries. The dislocations and twins formed due to cold work persisted even on overaging the alloy at 450°C for 16 h (Fig. 6b). It shows the onset of discontinuous precipitation at the grain boundaries of the matrix and also at the twin boundaries. However, when this alloy is overaged at 500°C for 32 h, coarse discontinuous precipitation is observed. The BF in Fig. 6c and DF in Fig. 6d show the coarse grains of discontinuous precipitate  $\beta$  in overaged alloy at 500°C for 32 h. When the alloy was severely deformed (90%), the dislocation and twin density increased significantly. Fig. 7a is a BF image of the 90% deformed alloy peak aged at 450°C for 15 min showing large twins and dislocation cells. The BF (Fig. 7b) and DF (Fig. 7c) show the presence of very fine ordered, metastable and coherent phase  $\beta^1$  Cu<sub>4</sub>Ti with bct structure and  $\alpha$ -matrix phase. The SAD (Fig. 7d) and its schematic (Fig. 7e) confirm the precipitate to be  $\beta^1$ , Cu<sub>4</sub>Ti phase.

An important observation in this investigation is that the T8 temper treatment (i.e. aging after severe cold work)



has resulted in the disappearance of the compositional modulations in this alloy, which were formed on water quenching during solution treatment in undeformed condition [9]. The TEM images in peak-aged condition after 50% deformation and 90% deformation (Figs. 6a and 7) reveal the absence of compositional modulations. This indicates that the role of compositional modulations is not present, unlike in undeformed alloy, in increasing the hardness and tensile properties after cold work and peak aging. Twinning mode of deformation is predominant in this alloy and is consistent with the earlier observations that Cu-Ti alloys having titanium above 4.0 wt.% deform by twinning [5, 6, 8, 16–18]. Therefore, dislocation cells, mechanical twinning and precipitation hardening due to  $\beta^1$ ,  $\text{Cu}_4\text{Ti}$  phase are the major strengthening mechanisms in the cold worked and peak aged Cu-4Ti-1Cd alloy.

The BF and DF images of the globular type discontinuous precipitate formed on overaging at 450°C for 16 h after 90% deformation are shown in Figs. 8a and b respectively. Fig. 8c and d are the SAD and its schematic respectively, which confirms the presence of  $\beta$ -precipitate. Fig. 8e shows the copious amount of fine equilibrium precipitate of  $\beta$ ,  $\text{Cu}_3\text{Ti}$  in the matrix in addition to high dislocation density. On overaging at 500°C for 32 h, the whole matrix was converted to a spheroidised structure. The BF (Fig. 9a) and DF (Fig. 9b) shows globular precipitation. The SAD (Fig. 9c) and its schematic (Fig. 9d) confirm the globular precipitate to be  $\beta$  phase in 90% deformed alloy over aged at 500°C for 32 h.

Overaging of the alloy in undeformed condition is associated with the formation of incoherent and equilibrium precipitate of  $\beta$ ,  $\text{Cu}_3\text{Ti}$  in the matrix and the discontinuous precipitation at the grain boundaries in lamellar morphology [9]. Overaging of the deformed alloy with 90% cold work brought out the morphological changes in discontinuous precipitation (DP), i.e. from lamellar form to globular form due to large strains induced by cold work.

When overaged at 500°C for 32 h, the recovery of matrix is reflected by decrease in hardness much below of that in solution treated condition. Extensive recovery of dislocations on overaging at 500°C is observed in the microstructures of this alloy (Fig. 6c and d, 8a and b and 9a and b). These TEM images also indicate the formation of globular discontinuous precipitation in the matrix. It was earlier reported that aging of solution treated and cold worked Cu-1.81Be-0.28Co alloy at 500°C produced faster rates of recovery and sub-grains resulting in the matrix [19]. The present results support this finding.

#### 4. Conclusions

The following conclusions have been drawn on the basis of the present study on age hardening behaviour of cold worked Cu-4Ti-1Cd alloy:

1. Substantial hardening occurred during the aging of deformed Cu-4Ti-1Cd alloy at 450°C. Hardness of 238 Hv, YS of 528 MPa and UTS of 754 MPa in solution treated condition of the undeformed alloy increased to 425 Hv hardness, 1037 MPa YS and 1252 MPa UTS on 90% cold work and peak aging at 450°C.

2. The ductility (%elongation) decreased drastically from 27% in solution treated condition to 1.2% in the alloy with 90% cold work and peak aging at 450°C.

3. The kinetics of aging got accelerated due to deformation and peak aging time decreased significantly (15 min) for 90% cold deformation and aging at 450°C.

4. The predominant strengthening mechanism of age hardening is the precipitation of an ordered, metastable and coherent  $\beta^1$   $\text{Cu}_4\text{Ti}$  phase in the matrix in addition to increased dislocation density and deformation twins.

5. Overaging of the alloy is associated with the formation of equilibrium phase of  $\beta$ - $\text{Cu}_3\text{Ti}$ . In addition, lamellar form of discontinuous precipitation at lower deformations and globular type at large deformations formed during overaging.

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