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ABSTRACT

THE EFFECT OF DEFORMATION TEMPERATURE ON
RECRYSTALLIZATION ON COPPER POLYCRYSTAL

by

Taekoo Lee

The influence of deformation temperature on recrystallization temperature was studied. To determine the tendency of this effect, flow stress curve method, microhardness measurements and microstructure developments were carried out. Contrary to one's expectation, recrystallization temperature decreases with increasing deformation temperature. This means that the more recovered the structure is, the more liable it is to recrystallize. Since recrystallization is a consequence of locallizing the stored energy, the more recovered structure becomes highly locallized in the stored energy. This fact suggests that due to the concurrent dynamic recovery, the structure becomes locally highly vulnerable to nucleation of recrystallization. This can be one key to understand dynamic recrystallization.

ACKNOWLEDGEMENTS

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1. INTRODUCTION

Hot working of metals is accompanied with softening process; recovery and recrystallization, which may be static or dynamic. The former is usually carried out by annealing in the absence of stress or strain, the latter takes place during high temperature deformation.

Recovery after cold working has been much investigated; the higher temperature of the annealing regime promotes climb and cross-slip which occurs either not at all or to a limited degree during deformation. In static recovery after hot working, there is no difference in the capability for climb and cross-slip. However, the initial substructure has already dynamically recovered extensively, compared with the cold worked state. It reduces the driving force and hence the rate of and refinement of recrystallization[1,2].

On the other hand, the process factors governing the recrystallization are; deformation temperature, annealing temperature, annealing time, strain rate, initial grain size and minimal strain. Many studies have already been establishedabout the interdependency of these quantities. The intention of this project was to elucidate the interdependency of these characteristic quantities, especially,

the effect of the deformation temperature on the recrystallization temperature at the corresponding flow stresses and how it is understood from the underlying physical processes. In this investigation, the stress was considered as a reference term rather than the strain, since the stress is a state parameter, while the strain, which depends on specimen history, is not. Also, the stress can be related to the microstructure in terms of the dislocation density, which is more appropriate for a discussion of the processes under consideration.

Three methods were selected to determine the recrystallization temperature; flow stress curve, using the intermediate interruption method, which will mention in detail later, microstructure evolution and microhardness measurement.

2. LITERATURE SURVEY

2-1 Cottrell - Stokes' Law

The stress-strain curves of a metal which are obtained at two different temperatures usually do not coincide. Fig. 1 shows schematically two such divergent curves measured at a constant strain rate at the temperature T_L and T_H. Cottrell and Stokes pointed out that this divergence may, in principal, at least, be the result of two distinctive contributions [3]. First, the amount of work hardening in each specimen with identical internal structures produced by a given plastic strain may depend on the deformation temperature. Secondly, for a given strain, the internal structure which has developed in each specimen as a result of plastic deformation depends on the deformation temperature. The term, internal structure, covers such features as small-angle boundaries, subgrains and cells, the density and configuration of dislocations, dislocation tangles, etc[3,4].

A method of seperating these effects is, referring to Fig. 1 , to deform to the point B at the temperature T_H and then to continue deforming at T_L, so obtaining the

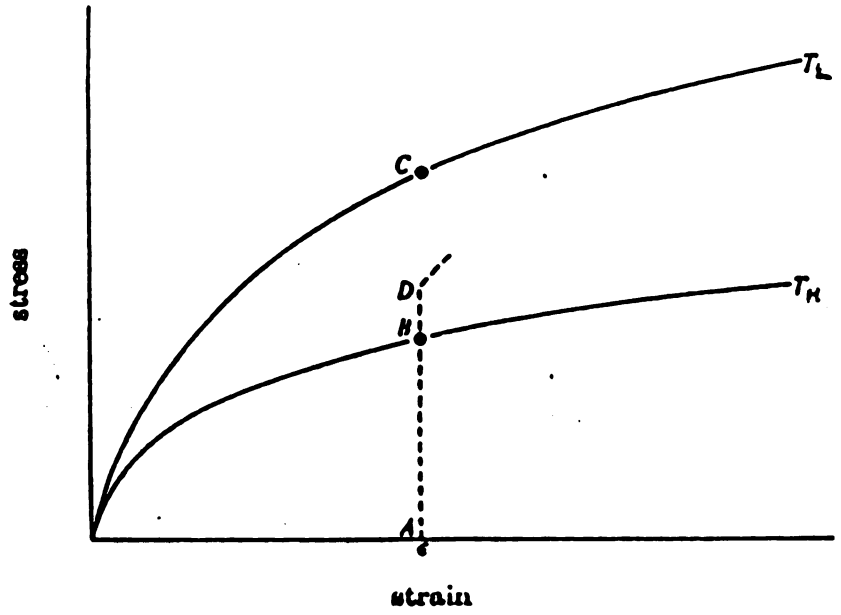


Fig. 1. Stress-strain curves (schematic) at two temperatures ($T_H > T_L$).

curve ABD. If aging and annealing effects are avoided between measurements of the flow stress at B and D, BD and DC are measurements of the reversible change of flow stress and of the difference in the levels of work hardening reached at the two temperatures, respectively. Cottrell and Stokes discovered an interesting relationship, the Cottrell-Stokes' law, which is to be noted that the measured ratio of flow stresses, i.e., AB/AD in Fig. 1, was independent of the strain at which the ratio was measured and of the thermal and mechanical history of the specimen [3,4,5]. The Cottrell-Stokes' law was found to be valid in the easy glide region of fcc crystals, whereas it may not be valid for bcc metals[4].

2-2 Dynamic recovery

In crystalline materials, during high temperature deformation the accumulated dislocations are continuously destroyed by two seperate processes; dynamic recovery and dynamic recrystallization. Dynamic recovery leads to the annihilation of pairs of dislocations, which gives rise to a lower work hardening rate than that in low temperature deformation, as well as to the formation of subgrains. In

high stacking fault energy materials, such recovery processes completely balance the effect of work hardening, that generates the dislocations, and lead to steady state flow[6,7,8], with the result that the dislocation density increases to an equilibrium value similar to that observed in cold worked and recovered metals[8]. In low stacking fault energy materials, by contrast, the dislocation density increases to very high level; eventually the local differences in density are high enough to permit the nucleation of recrystallization during deformation, so called, dynamic recrystallization[6,7,8].

The mechanism of dynamic recovery is related to that of static recovery, even though static recovery at the deformation temperature proceeds much more slowly than dynamic recovery [9,10,11]. There are two types of dislocation rearrangement processes in all materials; the process of cell-wall sharpening leads to dynamic recovery, that is present even at low temperature and the process of subgrain coarsening, whether by subboundary migration or dissolution, leads to dynamic recrystallization in low stacking fault energy materials[11]. The high temperature deformed dislocation structure shows cell structure and heavily tangled dislocation arrangements, however, the cell walls appear sharper, locally well defined subboundaries [11]. This substructure has a lower gradient in dislocation density from edge to center of grains than that at

ambient temperatures. In addition, as the dislocation density is lower by two orders of magnitude, there are fewer shear bands and other internal inhomogeneities than in cold working [7]. The higher the temperature of deformation, the larger the subgrains that are formed during high temperature deformation. As they increase in size, the subgrains contain fewer dislocations and have boundaries in which there are less dislocations arranged in more orderly arrays [12].

The process of dynamic recovery as it occurs during hot working cannot be considered as equivalent to the superposition of static recovery and cold working [12]. In fact, the small subgrains produced by hot working cannot be synthesized by cold working and annealing because of the intervention of recrystallization.

2-3 Static recrystallization after hot deformation

Recovery and recrystallization after cold working have been much studied; the higher temperature during the annealing promotes climb and cross-slip which occurs either not at all or to a limited degree during deformation. In static recovery during the annealing after hot working,

there is no difference in the capability for climb and cross-slip. However, the initial substructure has already dynamically recovered extensively [1]. In such circumstances, the recovered substructure reduces the driving force and hence the rate of recrystallization [1,12,13]. McQueen and Jonas have much investigated the effect of temperature on the rate of recrystallization, using the technique of interrupted compression test [1,14,15]. They have concluded that, at a higher temperature, more softening is achieved in a fixed time at the same strain, due to the increased thermal activation of static restoration (even though the stress and dislocation density at the end of the straining are lower), as shown in Fig. 2 [13]. In addition, the effect of temperature on the rate of recrystallization is that an increase in temperature of 50°C leads to a decrease in recrystallization time of about an order of magnitude and therfore to an approximate order of magnitude increase in recrystallization rate.

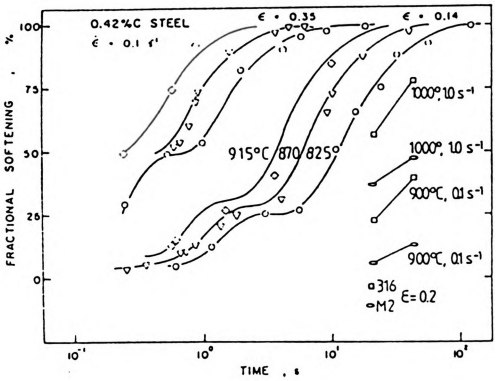


Fig. 2. The rate of static recovery and recrystallization in a 0.42 C steel after hot deformation to two strains.

3. EXPERIMENTAL PROCEDURE

The method of intermediate interrupting employed in the present study , where the recrystallization temperature after interrupting and annealing was measured at three different deformation temperatures, is briefly described here. The method is based on the principle that recrystallization restores the mechanical properties to the initial state. Variation in the flow stress with the temperature was measured over a range of strains and temperatures to determine the reference stress at each deformation temperature by means of Cottrell-Stokes' law. Samples were loaded to the each reference stress at a constant true strain rate, $2x10^{-4}$/sec, in compression or in tension, at each deformation temperature, and then unloaded. After annealing at $300^{\circ}C$ for 1h in the vacuum furnace, a metallography and microhardness measurement were done. Again, the samples were reloaded to the same stress at the same strain rate as before. The same procedure was repeated with the increasing annealing temperature, in the steps of $50^{\circ}C$, until complete restoration in stress-strain curve was shown.

3-1 Specimen Preparation

3-1-1 Mechanical Testing Specimens

Compression samples were machined into right cylinders, 3.2 mm in diameter and 4.5 mm in height, and tension samples, whose shape and dimensions are shown in Fig. 3, were machined from 99.99% pure polycristalline copper cylindrical bar with 1/2". The specimens were annealed at 600°C for 6h in a vacuum furnace at a pressure of approximately 10^{-5}-10^{-6} torr and then cooled in the furnace. The resulting grain size, measured by mean linear intercept method, was 30 μm. And then, they were etched in 50% HNO_3 by approximately 0.1 mm from the surface, to remove any surface irregularity. Finally, all samples were chemically polished in a solution of equal parts of nitric acid, phosphoric acid and glacial acetic acid in order to avoid any potential sites for recrystallization during annealing.

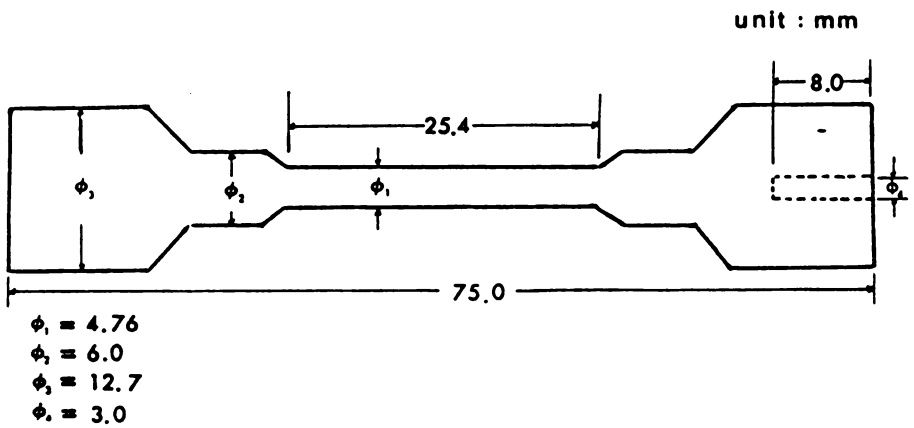


Fig. 3. Geometry and dimensions of tension sample

3-1-2 Optical Microscopy Specimens

The samples deformed at 3 different temperatures were cut into two pieces each, by diamond blade, after each deformation at three different temperature. The cut samples were etched in 50% HNO_3 by approximately 0.1mm from the surface. Then, each sample was annealed at each annealing temperature, which is mentioned before. Microstructure was taken from the cut face of the annealed sample. Specimens were mounted in lucite and mechanically ground with abrasive grit papers of 240 grit to 600 grit and polished on a cloth, using aluminum oxide of the size from 5 μm to 0.3 μm to get a scratch-free surface. Finally, they were etched in the following etchant:

Dist. water or Ethanol (95%)	100-120 ml
Hydrochloric acid (1.19)	20-50 ml
Iron(III) chloride	5-10 ml

3-2 Mechanical Testing

Axisymmetric, isothermal tests were conducted on a computer-controlled electro-mechanical screw type Instron testing machine with a 500 kg tension-compression reversible load cell. Pull rods and button head grips were designed and machined out of 310 heat resistant stainless steel. To avoid oxidation during high temperature testing, all tests were carried out in a cylindrical tube which was made of Invar and which stayed with a gap of 0.5 mm from the upper pull rod. The lower end of the tube was tightly fit with the lower pull rod due to the difference in thermal expansion between Invar ring and lower pull rod. During the test the specimens were kept in this environmental chamber under an atmosphere of 90% N_2+10% H_2 to prevent oxidation. The arrangement of the hot chamber is schematically given in Fig. 4. The temperature was recorded by a chromel/alumel thermocouple, which kept in contact with the lower grip in compression test, and with the specimen in tension test. The temperature of the specimen was kept constant within $\pm 1^{\circ}C$ during the deformation. The specimens were loaded up to 5 kg, using "Load" mode in cycle control of Instron, during heating. In the compression tests the cross head speed was chosen 0.002 inch/sec and 0.01

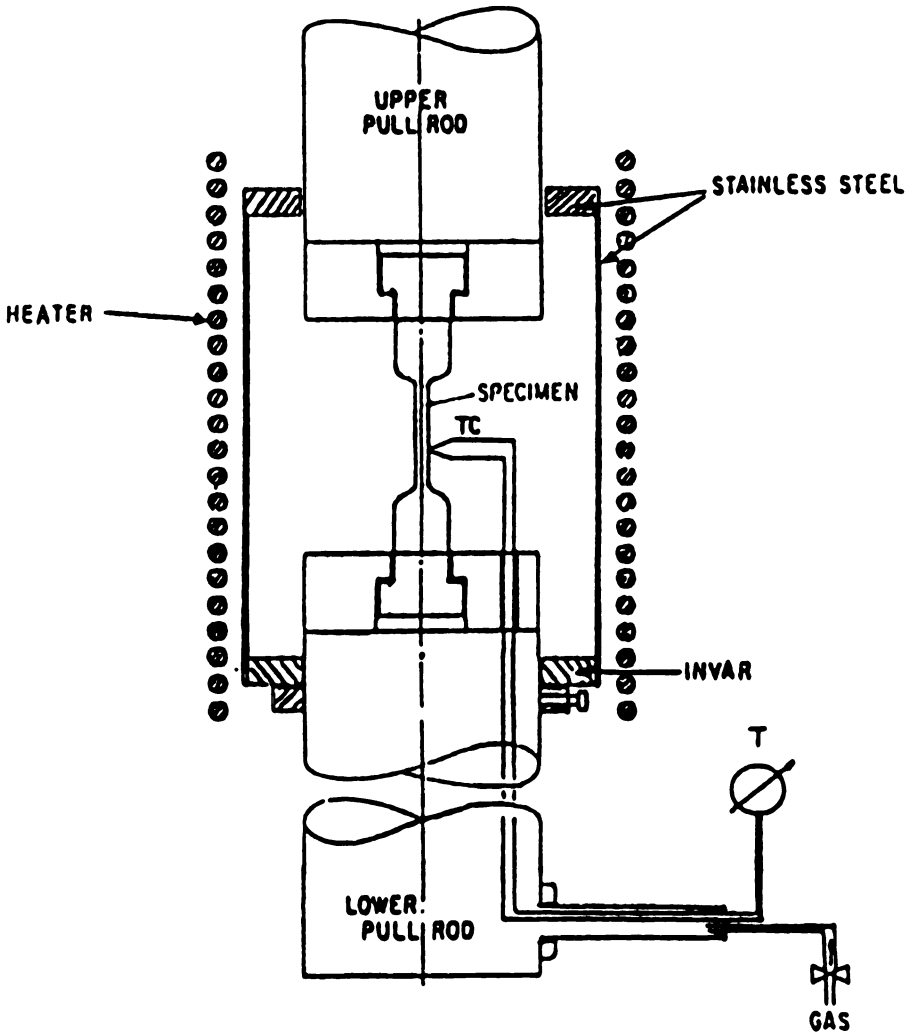


Fig. 4. Arrangement of the high temperature mechanical testing setup.

inch/sec in tension tests, so that inital strain rate was approximately $2x10^{-4}$/sec.

3-3 Microhardness Test (Vickers Hardness)

The microhardness test was carried out on Micromet II Digital Microhardness Tester manufactured by Buehler LTD. Each specimen was indented at 25 points distributed at random, and the values were averaged. Applied load, loading time and loading speed were chosen: 0.3 kg, 20 sec and 50 m/sec, respectively.

3-4 Method Of Determination of reference stresses

Change in the flow stress of crystals with the change in the deformation temperature was measured over a range of strains and temperatures to determine the reference stress at each deformation temperature by means of Cottrell- Stokes' law. A specimen was deformed to an arbitrary stress prior to the dynamic recrystallization at

400°C. After unloading, the specimen was cooled in situ to 300°C as fast as possible. The specimen was reloaded and deformation was continued right beyond the stress, at which flow stress was starting, at the strain rate which was employed for predeforming. Above procedure was repeated at 200°C, 100°C and room temperature on further in situ cooling. In order to make sure the accuracy of the results,these procedures were repeated with 5 different specimens: two of them in tension test and rest of them in compression test. To obtain sufficient deformation conditions for SRX, $0.9\sigma_R$[σ_R :DRX stress at 400°C] was taken as reference stress at 400°C. Cottrell- Stokes' ratios were calculated from above results. Equivalent stress at each temperature was computed, using $0.9\sigma_R$ and Cottrell- Stokes' ratios.

4. EXPERIMENTAL RESULT

4-1 The effect of deformation temperature on the flow stress of copper polycrystals

A set of five serially conducted stress-strain curves are shown in Fig 5, obtained from tests carried out on high purity polycrystals of copper by cycling between 400°C and room temperature, as mentioned in the previous chapter. Changes in flow stress produced by changes from 400°C to room temperature, during the tension or compression tests on the crystal at various strains, are given in the Table 1. These stress-strain curves show that the flow stress increases with decreasing deformation temperature, irrespective of the amount of prestrain. In addition,the ratio of $\sigma_{300}/\sigma_{400}$, $\sigma_{200}/\sigma_{300}$, $\sigma_{100}/\sigma_{200}$ and σ_{RT}/σ_{100} was calculated at five different strains, and are shown in Fig. 6. The average of the each ratio was : 1.02, 1.03, 1.046 and 1.041, respectively. These results indicate that, as the deformation temperature decreases, the increment in the flow stress gets larger. Fig.6 illustrates this fact. Finally, the results of this experiment indicated that the ratio of the flow stress at

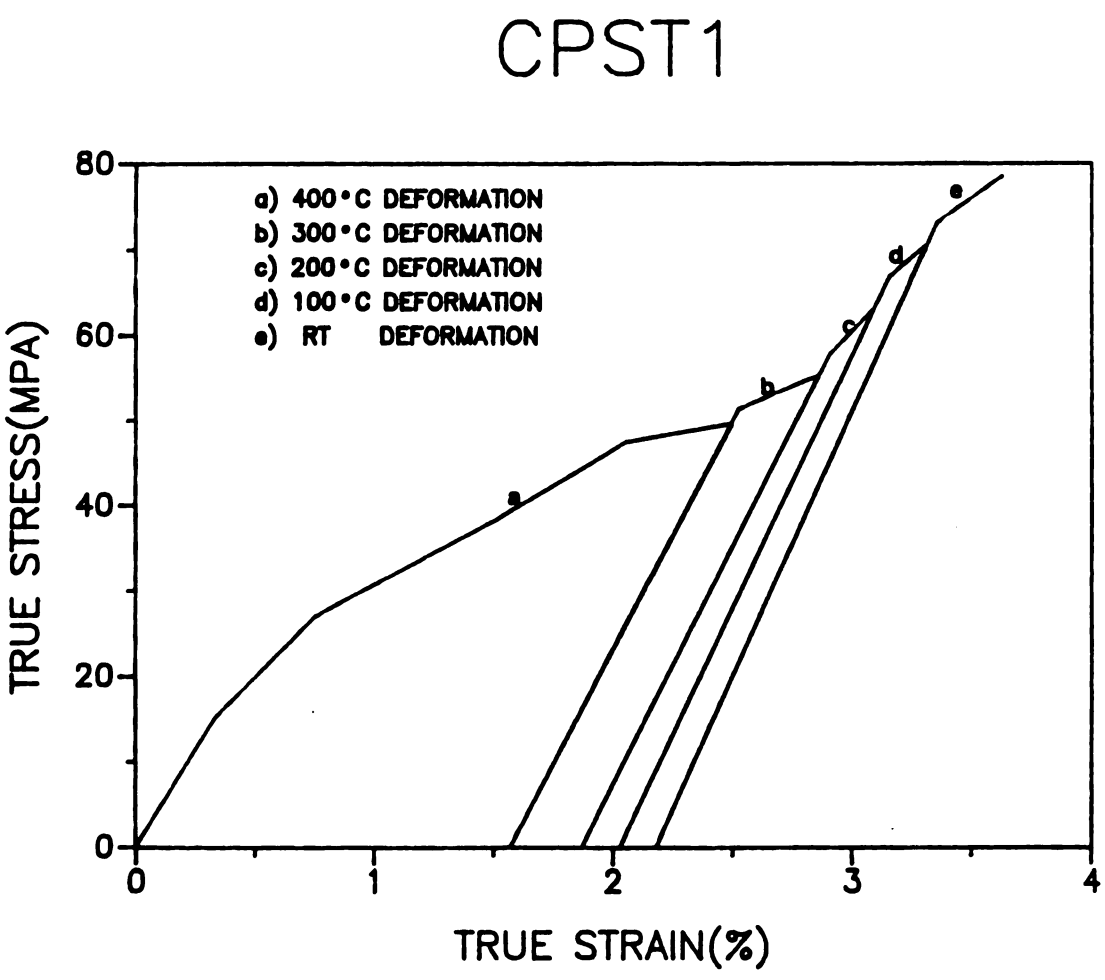
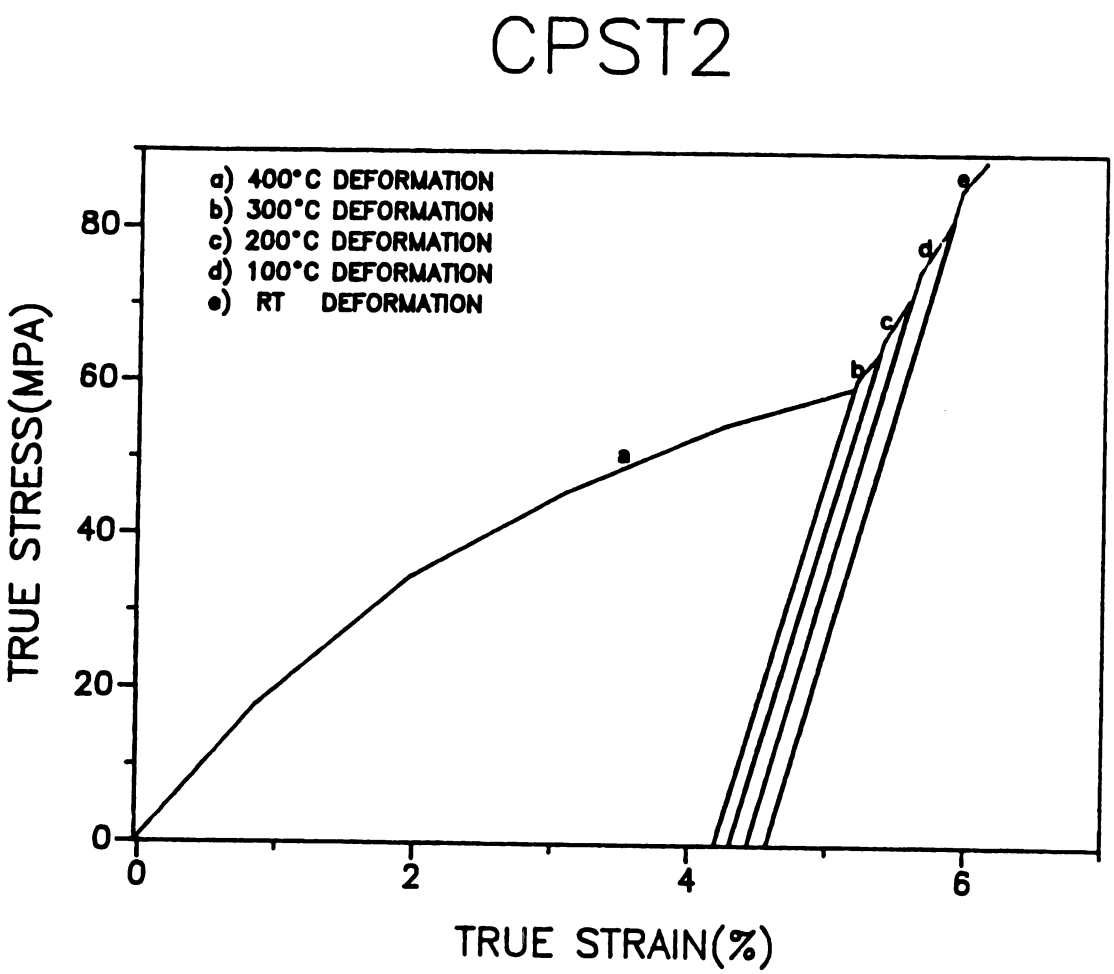
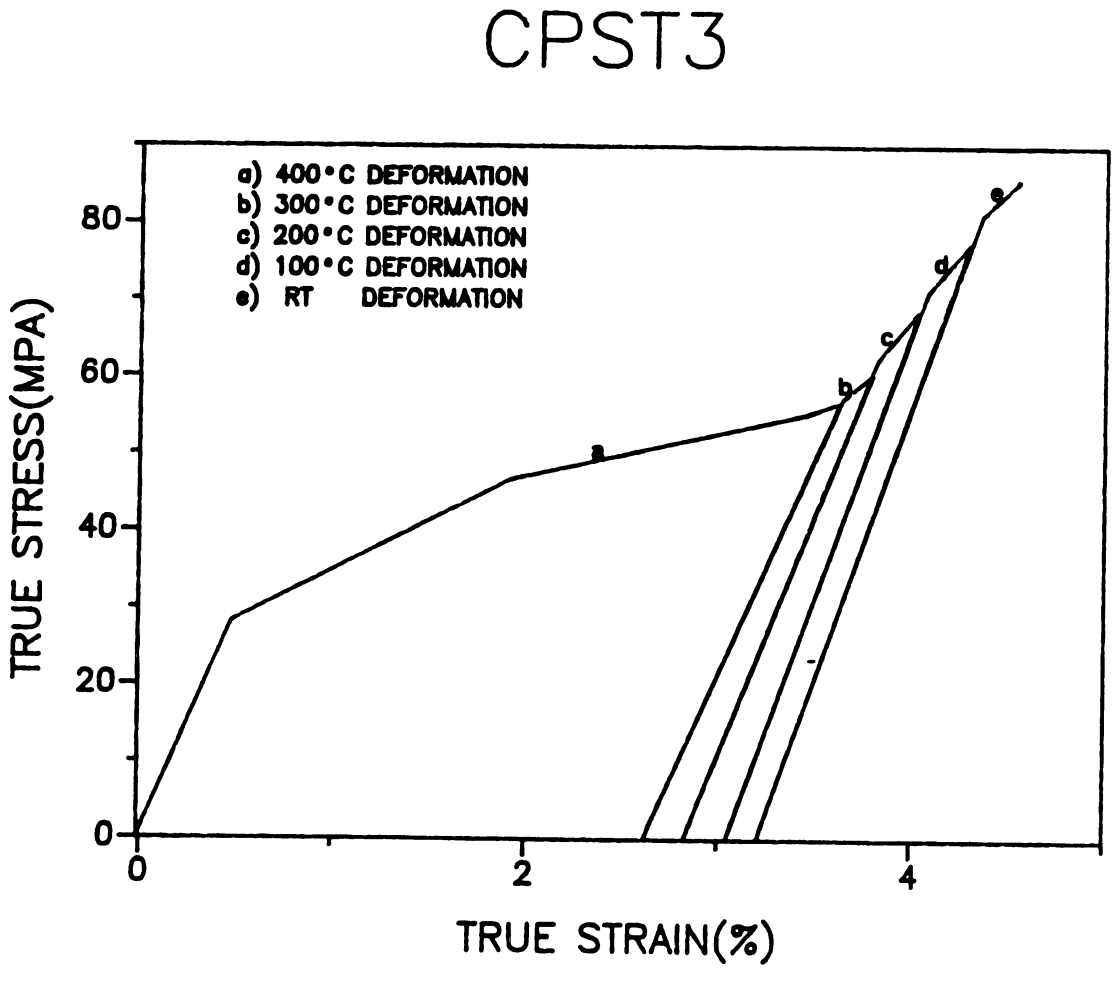


Fig. 5. A set of serially conducted stress-strain curves obtained from cycling tests with changing the temperature from 400°C to RT.

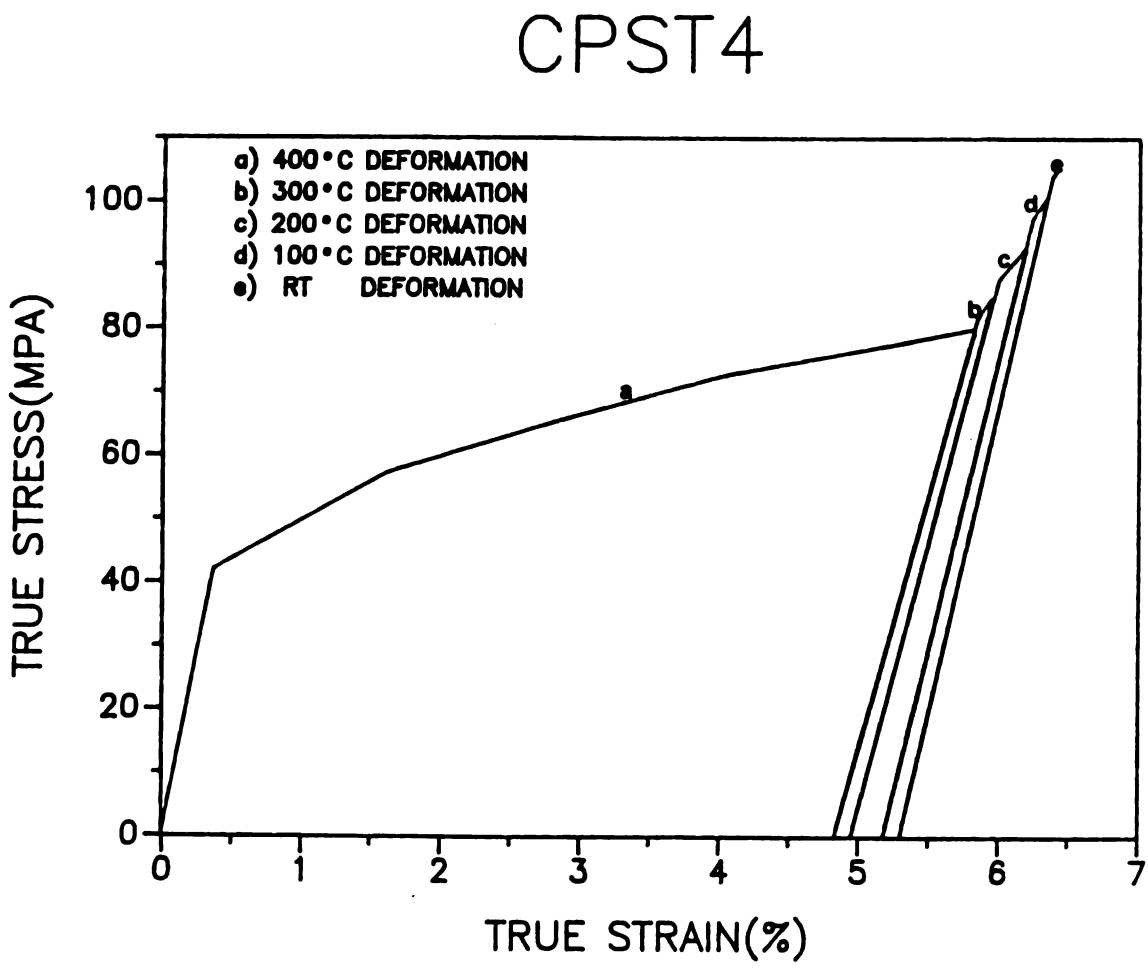
a) prestrain (%) : 2.5



b) prestrain (%) : 5.19



c) prestrain (%) : 3.7



d) prestrain (%) : 5.25

Table 1. Changes in flow stress with temperature change

a) TEST NAME : CPST1

PRESTRAIN(%): 2.5

TEST MODE : COMPRESSION

σ^1: new yield stress

σ^2: unloading stress

	400°C	300°C	200°C	100°C	RT
σ^1		50.9	57.8	66.7	73.0
σ^2	49.7	55.3	63.0	70.4	

b) TEST NAME : CPST2

PRESTRAIN(%): 5.19

TEST MODE : COMPRESSION

	400°C	300°C	200°C	100°C	RT
σ^1		60.5	65.8	74.7	85.2
σ^2	59.2	64.0	71.0	81.4	

c) TEST NUMBER : CPST3

PRESTRAIN(%): 3.7

TEST MODE : COMPRESSION

	400°C	300°C	200°C	100°C	RT
σ^1		57.7	62.3	71.2	80.9
σ^2	56.9	60.4	68.3	77.3	

d) TEST NUMBER : CPST4

PRESTRAIN(%): 5.25

TEST MODE : TENSION

	400°C	300°C	200°C	100°C	RT
σ^1		81.6	87.8	97.1	103.5
σ^2	80.1	84.9	92.8	100.5	

e) TEST NUMBER : CPST5

PRESTRAIN(%): 7.38

TEST MODE : TENSION

	400°C	300°C	200°C	100°C	RT
σ^1		81.4	87.6	95.6	103.6
σ^1	80.0	85.6	91.4	99.1	

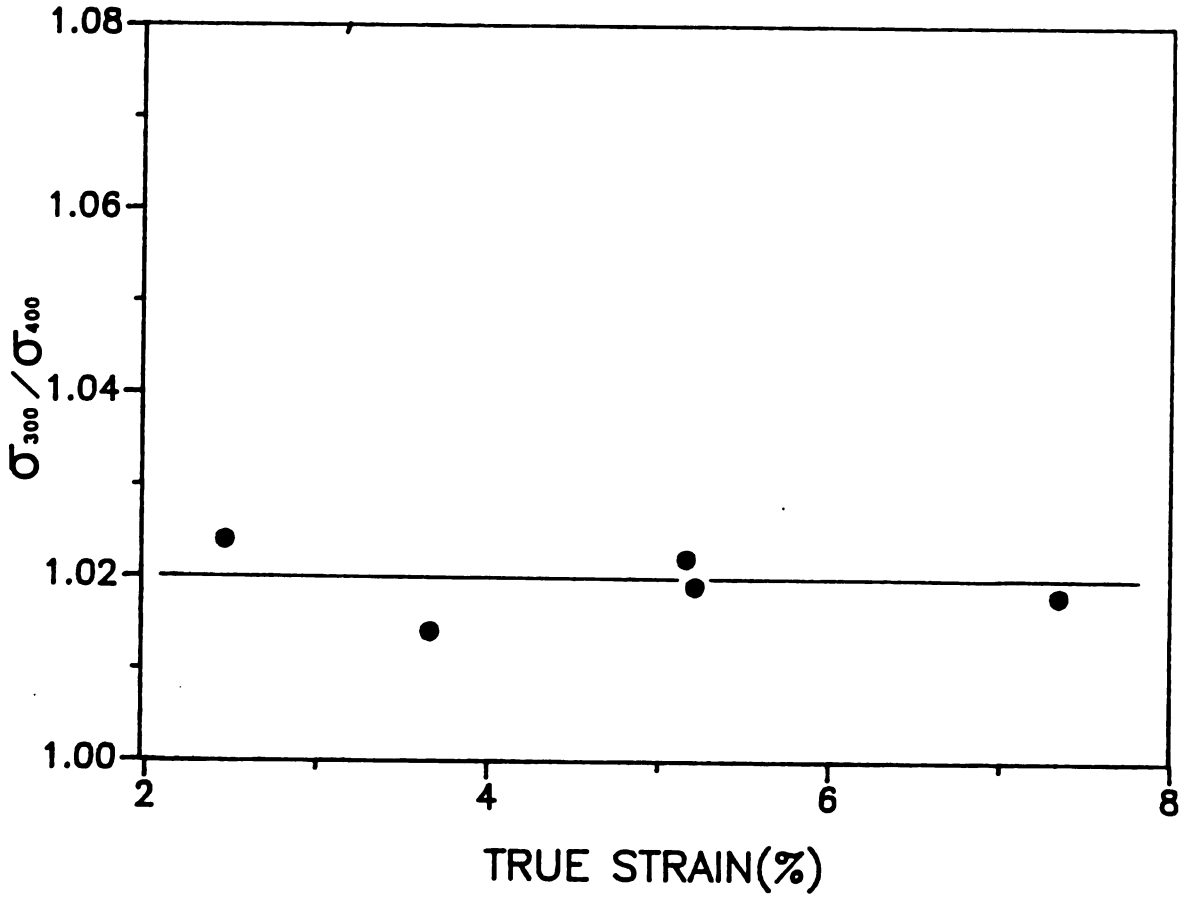
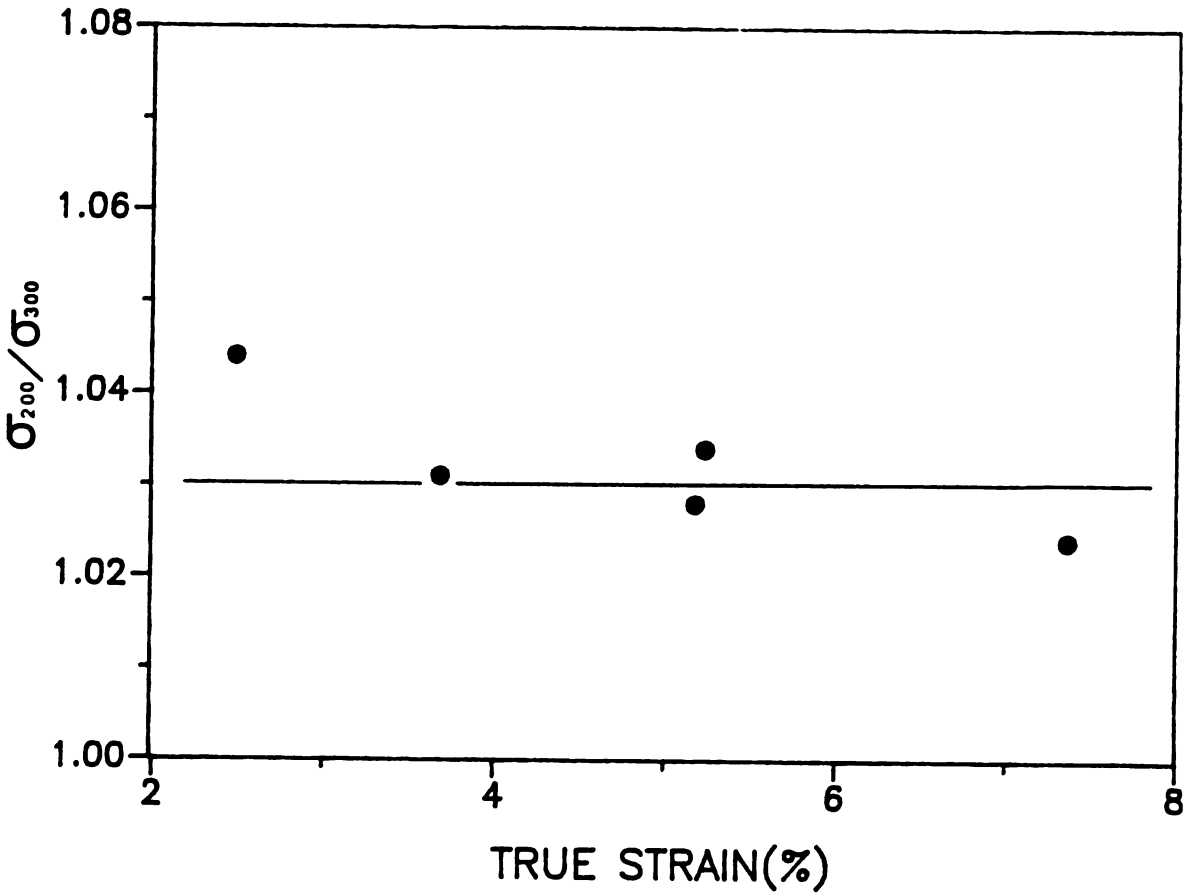
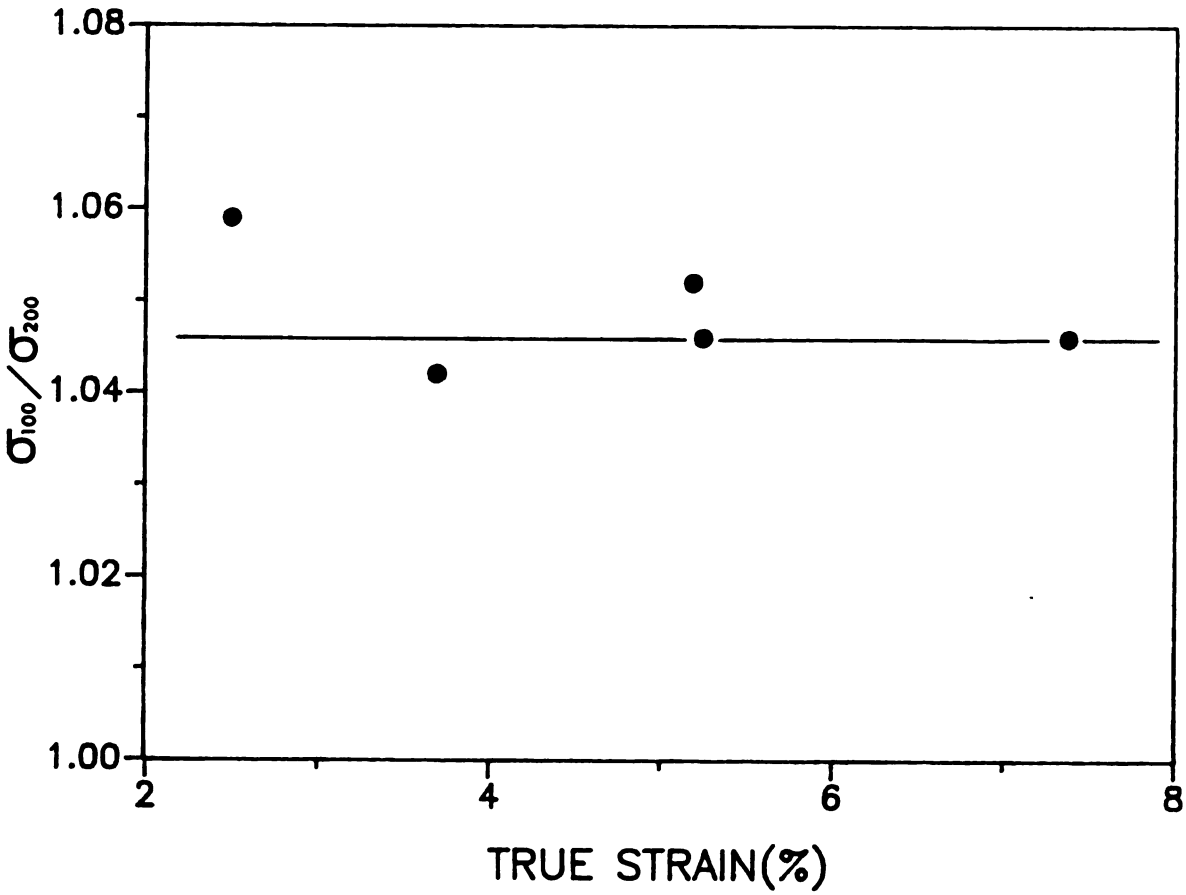


Fig. 6. Cottrell-Stokes' ratioes at various temperatures, calculated at five different prestrains.

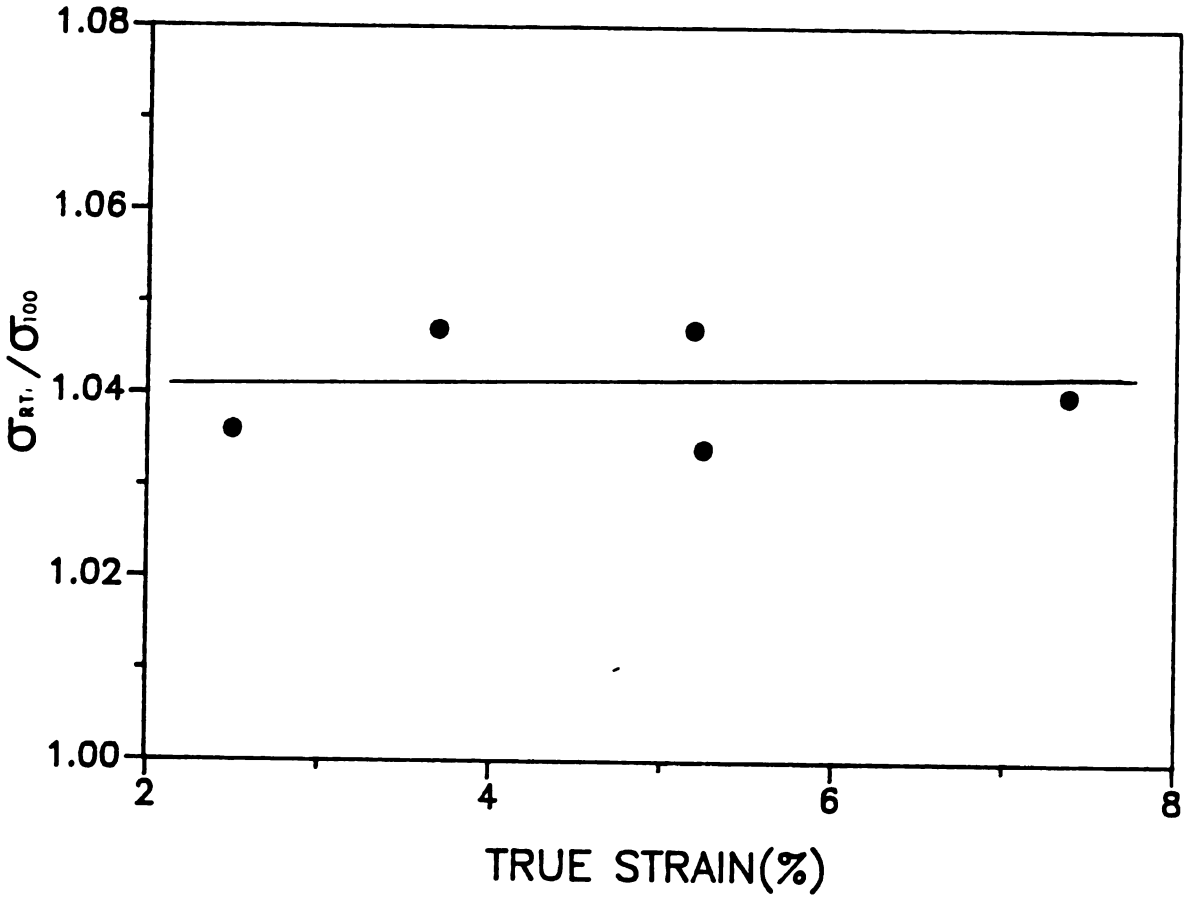
a) σ_{300} / σ_{400}



b) σ_{200} / σ_{300}



c) σ_{100} / σ_{200}



d) $\sigma_{RT} / \sigma_{100}$

room temperature, relative to that at 400°C (Cottrell-Stokes' ratio), is 1.144.

In a similar study of temperature-dependence of flow stress on copper single crystals, Schmid indicated that the Cottrell-Stokes' ratio calculated for the flow stress at room temperature to the flow stress at 400°C was 1.05. On the other hand, Cottrell and Stokes, who have already observed this ratio on OFHC copper single crystal at various elongations, have obtained these ratios for the values of the flow stress measured at 200°C to that at -183°C, as lying between the range of 0.783 - 0.853 [5].

4-2 Determination of reference flow stress at different deformation temperatures

A study of the influence of grain size on the flow behavior of pure polycrystalline copper was also carried out during the course of this investigation. Fig. 7 illustrates the completely dynamically recrystallized (steady state) flow curves for specimens having different grain sizes at a temperature of 400°C. The results obtained by Deshpande [16] represent considerably difference from the results obtained during the course of the present investigation. This can be attributed to the fact that

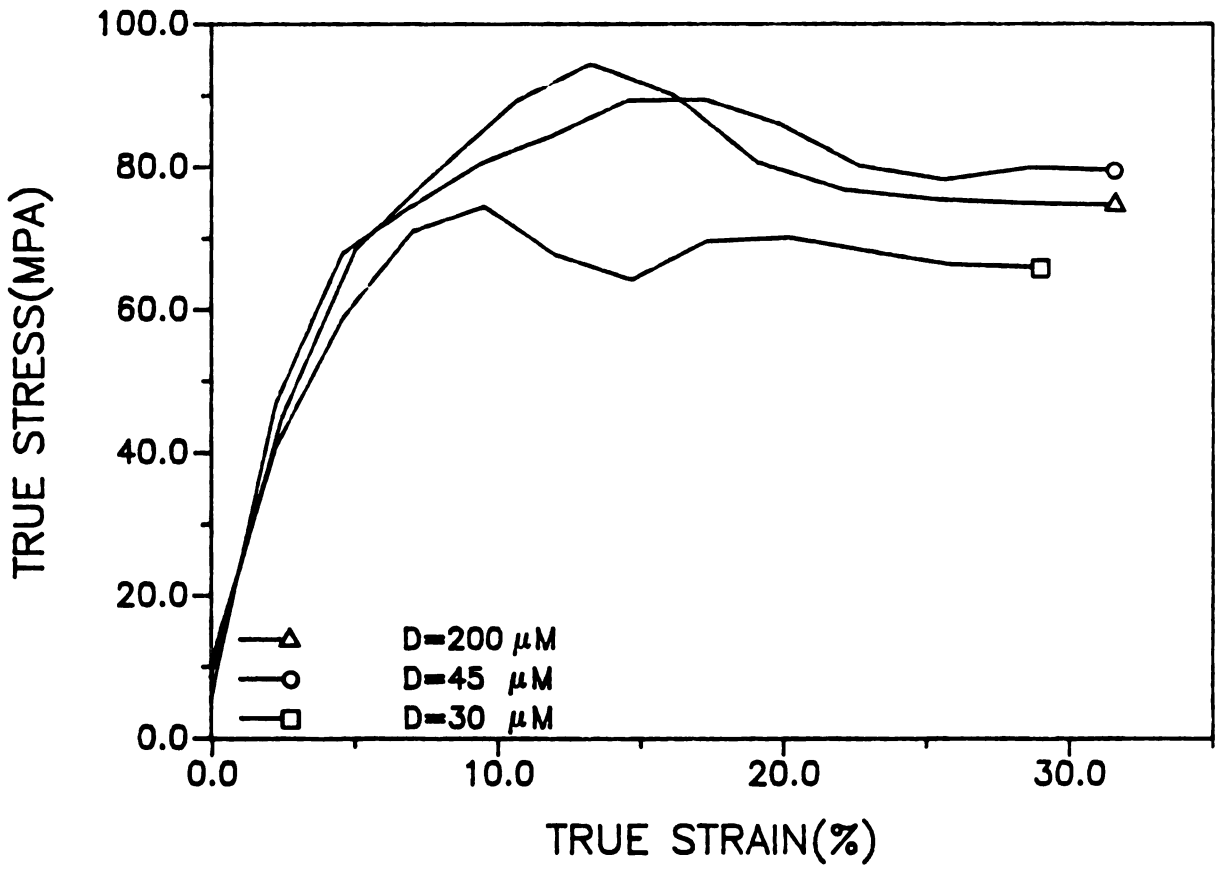


Fig. 7. The completely dynamically recrystallized (steady state) flow curves for specimens having different grain sizes at a temperature of 400°C.

Deshpande annealed his specimens in air, unlike in the present case, in which the specimens were annealed in vacuum. As a result of annealing in air, it is possible that the specimens by the former, were hardened due to the impurities formed due to oxidation. Also, Petkovic et al have observed the different flow curves of polycrystalline copper at 450°C due to difference in purity of the specimens[14].

A grain size of 30 μm was chosen in this study and the DRX stress(σ_R) measured was 74 MPA at 400°C. In this investigation, the critical stress required for the specimens to recrystallize statically was chosen as $0.9\sigma_R$ which was also taken as the reference stress at a temperature of 400°C for further investigation. Table 2 indicates the reference stresses, which are normalized to an equivalent state for various temperature, using the Cottrell-Stokes' ratios.

Table 2

Temperature (°C)	400	300	200	100	RT
Flow Stress (MPA)	66.6	67.9	70.0	73.2	76.2

4-3 The effect of the deformation temperature on the static recrystallization temperature

4-3-1 Mechanical property observation

As stated earlier, interrupted tests were conducted, at temperature of 400°C, 200°C and room temperature. The initial true strain rate was approximately 2×10^{-4}/sec. The stress strain curves, which have gone through the course of interrupted tests at each deformation temperature, are shown in Fig. 8. The curves exhibit the new yield points following annealing after each interruption. As the annealing temperature was getting higher, the amount of the drop in flow stress also increased. At last, the complete restoration of stress-strain curve was observed. For 400°C deformation, fully restored curve appeared after annealing at 450°C. For 200°C deformation and room temperature deformation, it appeared after annealing at 500°C. These results represent that fully static recrystallization occurred at the annealing temperature of 450°C for 400°C deformation and at 500°C for 200°C deformation and room temperature deformation. Another evidence of above results

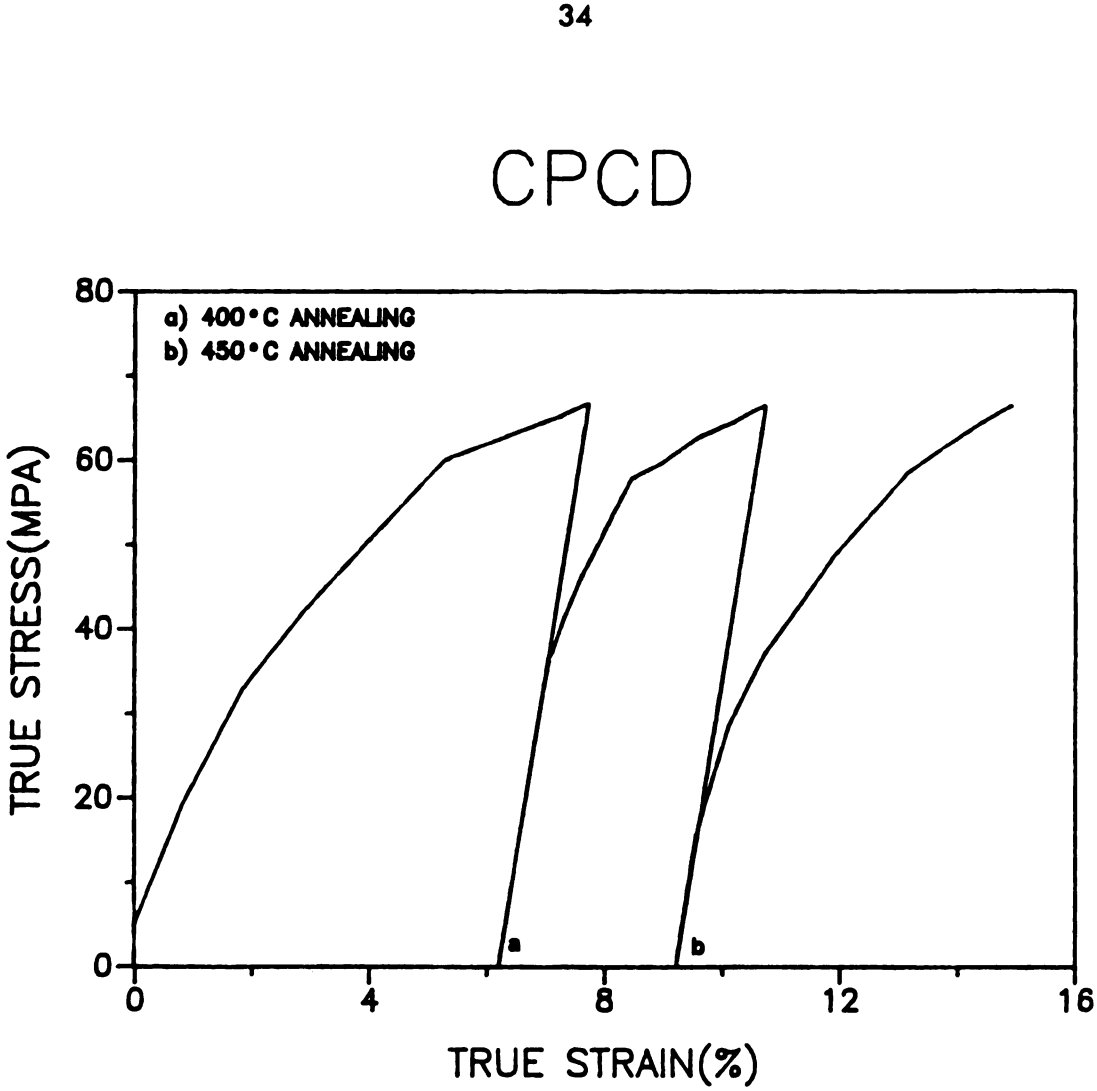
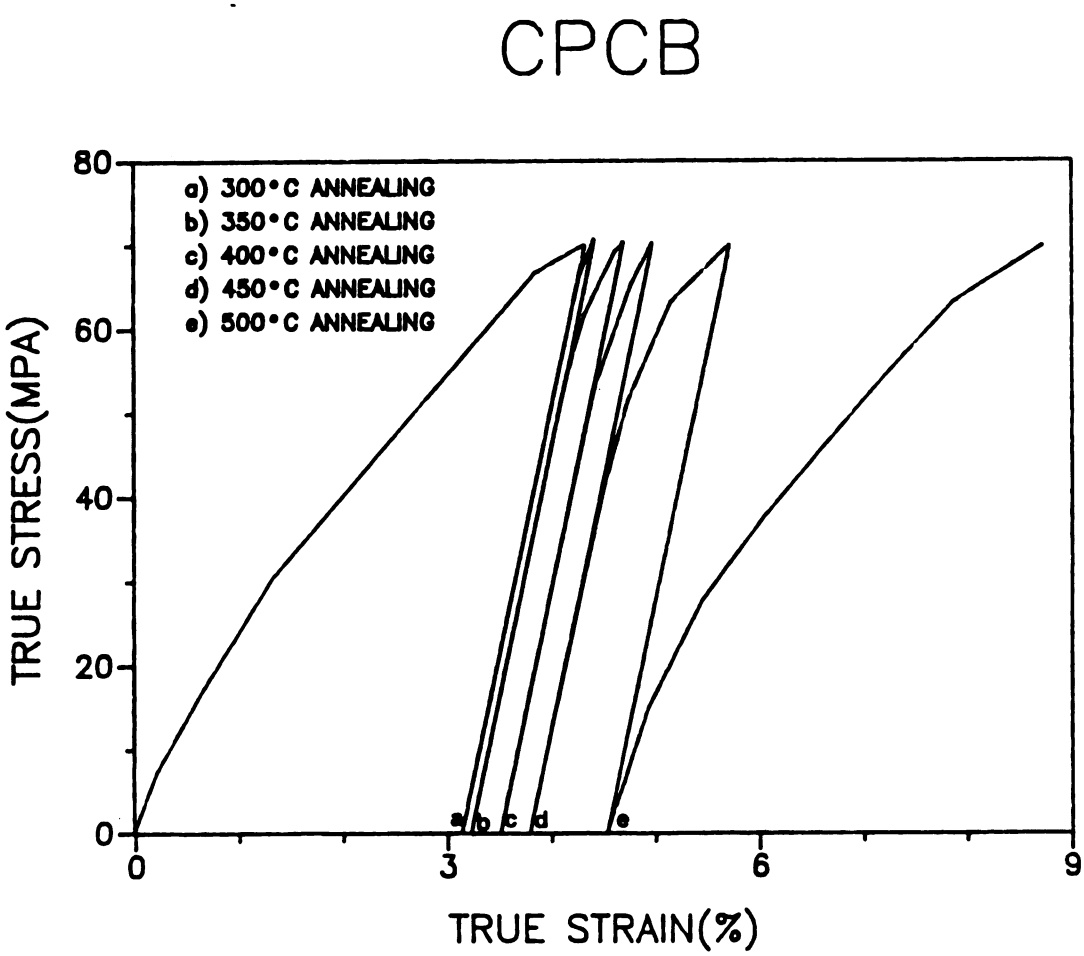
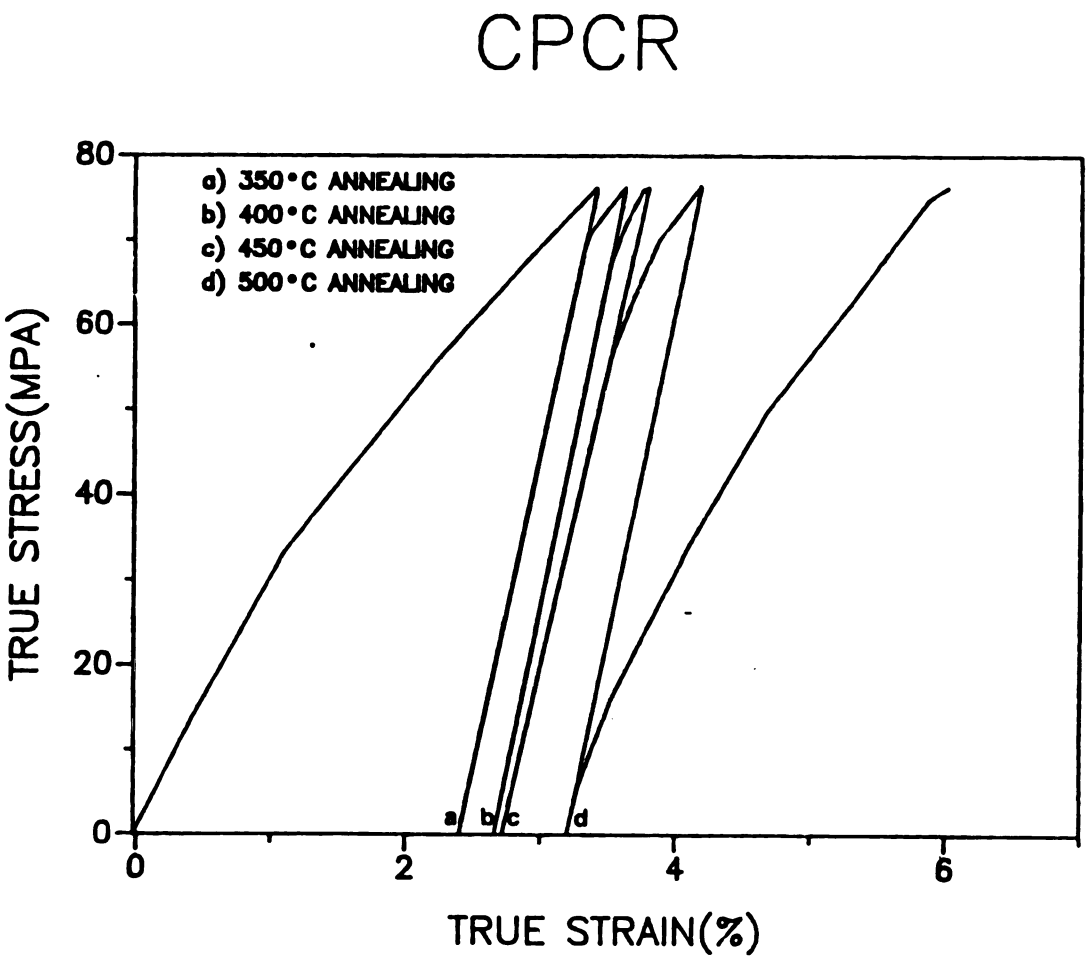


Fig. 8. The stress-strain curves through the course of interrupted tests with increasing annealing temperature

a) for 400°C deformation temperature



b) for 200°C deformation temperature



c) for RT deformation temperature

is shown in fractional softening curves, Fig. 9. The fractional softening is calculated from the drop in flow stress following annealing after each interruption.

$$F.S = (\sigma_m - \sigma_r)/(\sigma_m - \sigma_o)$$

Here, F.S is the fractional softening during annealing, σ_m is the stress just before unloading, i.e., the reference stress in present study. σ_o and σ_r are the initial flow stresses recorded during prestraining and reloading, respectively. The curves in Fig. 9 exhibit the same generally sigmoidal form and illustrate that, at the same annealing temperature, the value of fractional softening is increasing with the increasing deformation temperature. In addition, for 200°C deformation and room temperature deformation, fractional softening of 98.5% and 91.4% at the same annealing temperature of 500°C, was shown in the factional softening curves. This fact implies that, although fully restored curve appeared at the same annealing temperature for both cases, recrystallization temperature for room temperature deformation is higher than that for 200°C deformation.

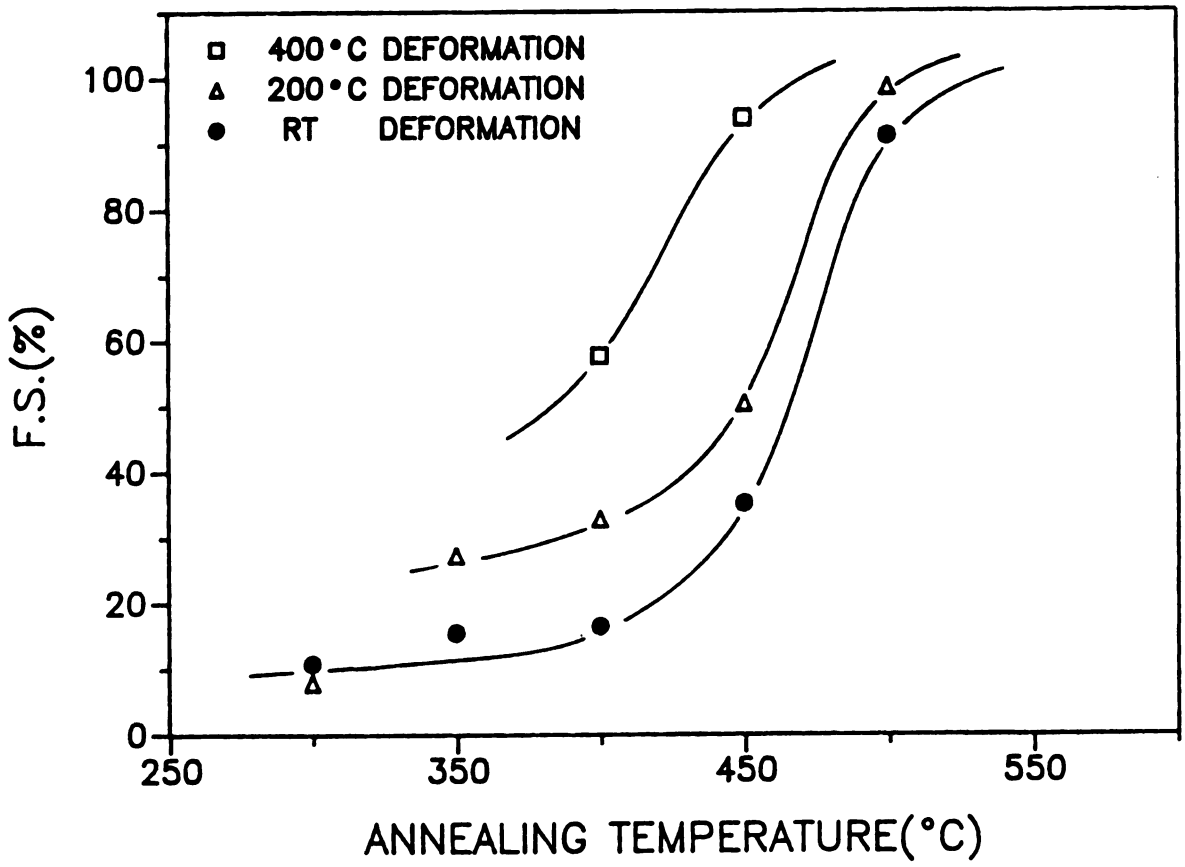


Fig. 9. The effect of temperature of deformation on the fractional softening at a fixed annealing temperature

4-3-2 Microhardness measurement

Change in the microhardness with the increasing deformation temperature is shown in Fig. 10. The minor change in the microhardness at the front part and the rear part of the curves might result from recovery and grain growth, respectively. However, the major change in the microhardness at the middle part of the curves can be regarded as simultaneous recrystallization. These curves illustrate that for 400°C deformation, this major change in the microhardness starts in the annealing temperature range of 350°C - 400°C and ends in the range of 450°C - 500°C, and for room temperature deformation, it starts in the range of 450°C - 500°C and ends up at 550°C. Also, the microhardness curve for 200°C deformation represents the intermediate values of the temperature range of onset of the major change and of completion of it. These results indicate that, as the deformation temperature increases, the onset temperature for recrystallization decreases and the temperature for complete recrystallization also decreases.

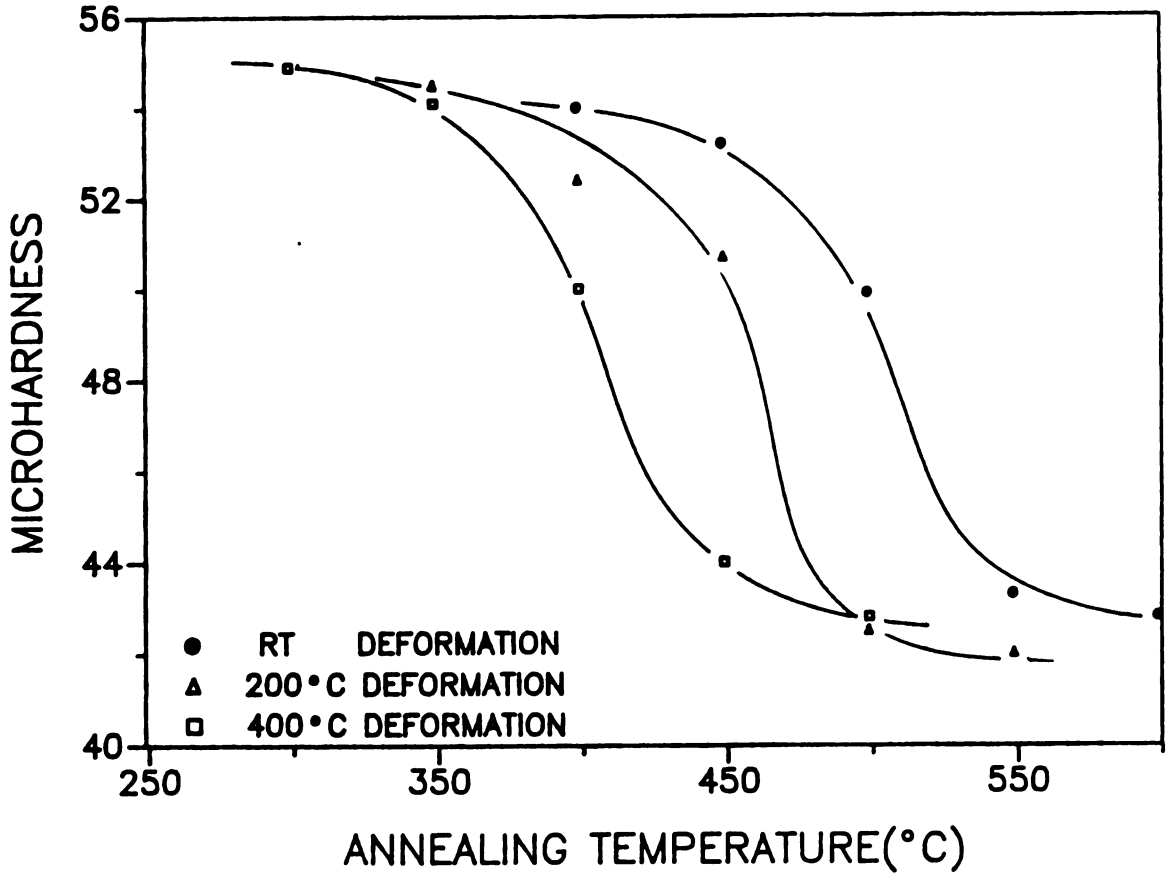


Fig. 10. The microhardness curves with changing the annealing temperature at different deformation temperature.

4-3-3 Microstructure observation

The progress of static recrystallization was determined from the optical examination of specimens at the end of annealing after each interruption. A series of structures, which was selected at the increasing annealing temperature for deformation at 400°C, 200°C and room temperature, is presented in Fig. 11, Fig. 12 and Fig. 13, respectively. For deformation at 400°C, recrystallization was observed to start to occur in the annealing temperature range of 350°C - 400°C and to complete after 450°C [Fig. 11]. A sample deformed at 200°C contained a few new grains after annealing at 450°C and completely recrystallized after annealing at 550°C [Fig. 12]. For deformation at room temperature, onset of recrystallization was observed after annealing at 450°C and full recrystallization after annealing at 550°C [Fig. 13]. Roberts [18] suggested that the statically recrystallized grain size is seen to increase steadily as recrystallization progresses, even for grains in the middle of recrystallized areas (i.e. very rapid grain growth is involved in addition to grain-boundary migration driven by a dislocation density difference). Also, the sample was annealed at 500°C for 1 h without deformation, however, there was no structure

change at all. This fact can be an evidence that the new large grains must be the recrystallized grains.

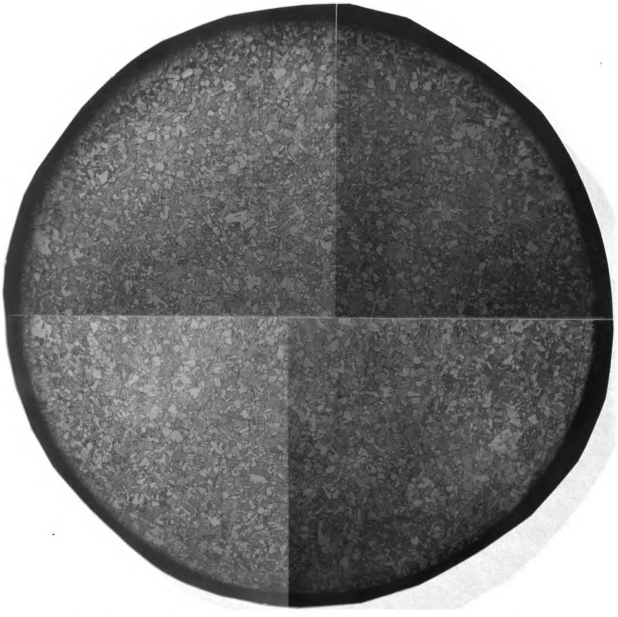
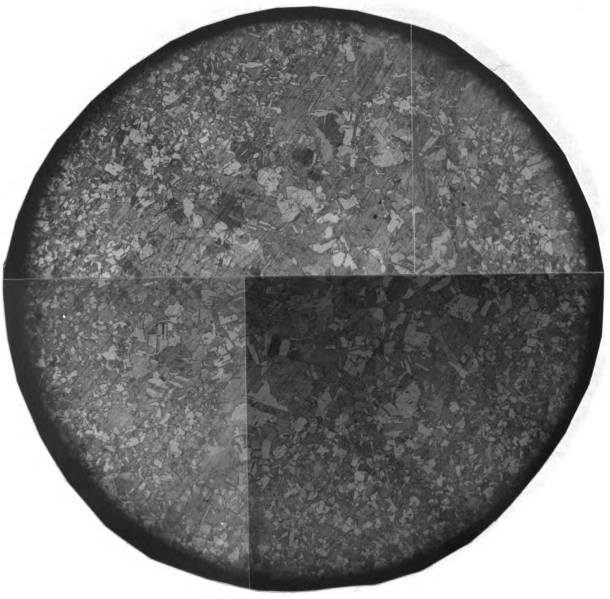
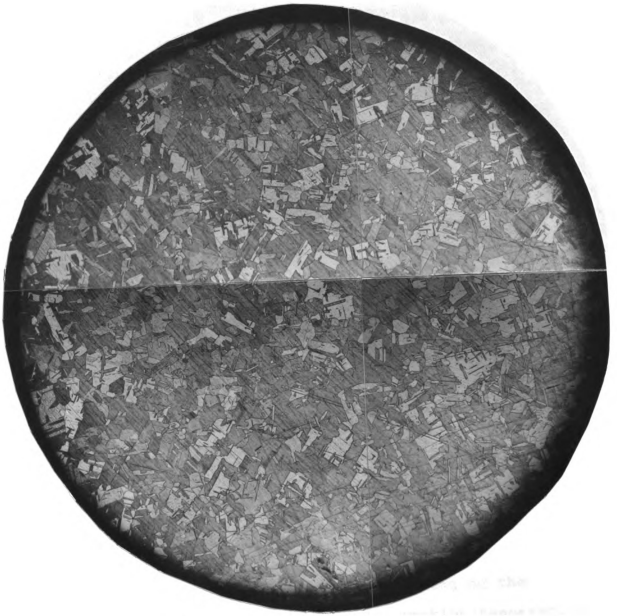


Fig. 11. Microstructure evolution with changing the annealing temperature at deformation temperature of 400°C.(X 50)
a) after annealing at 350°C



b) after annealing at 400°C



c) after annealing at 450°C

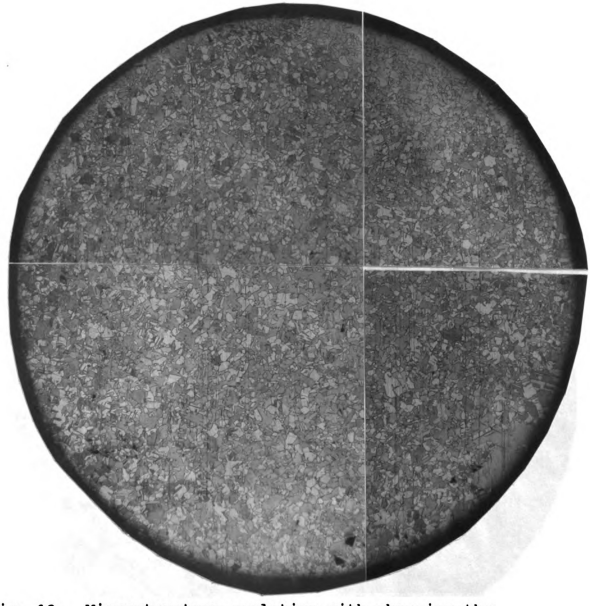
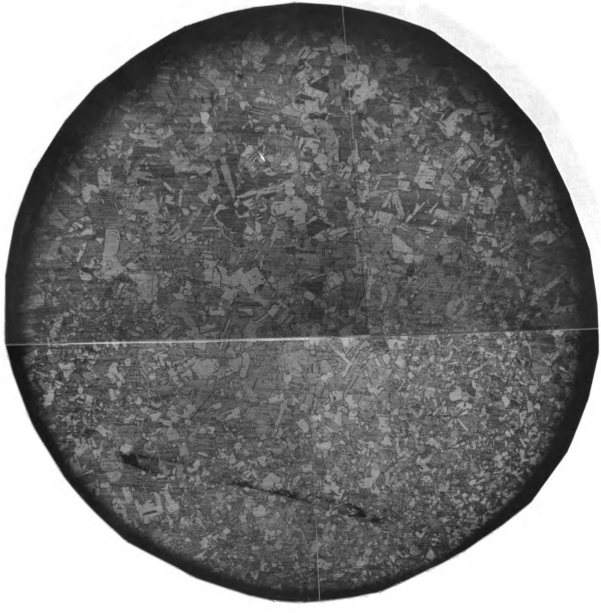


Fig. 12. Microstructure evolution with changing the annealing temperature at deformation temperature of 200°C (X 50)

a) after annealing at 450°C



b) after annealing at 500°C

c) after annealing at 550°C

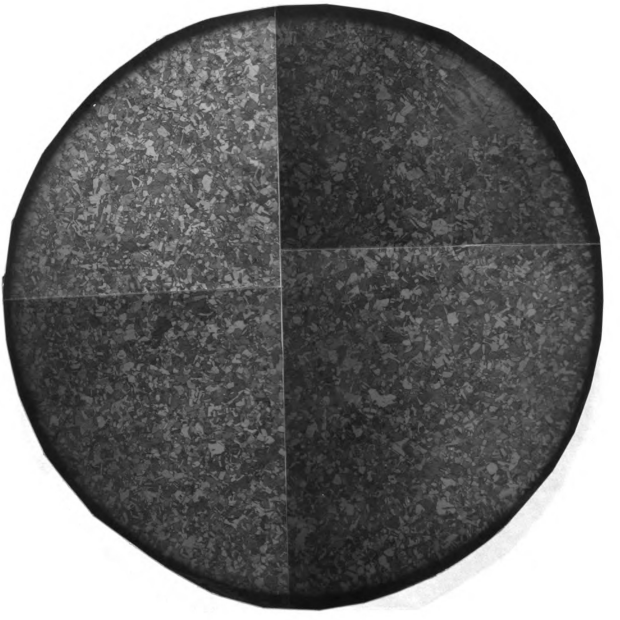
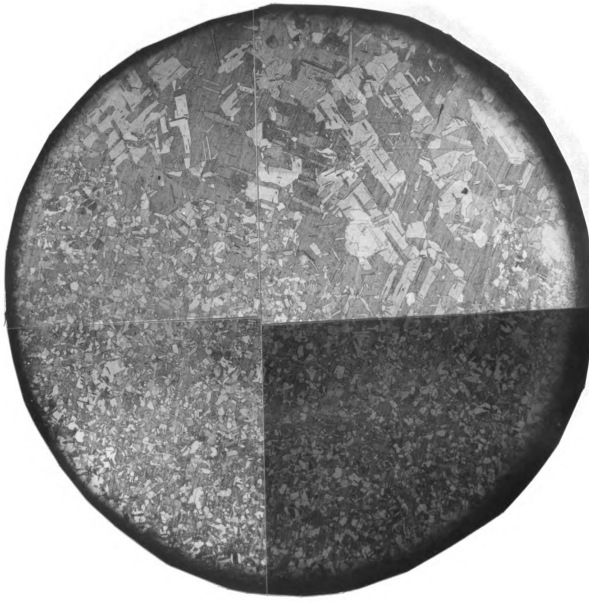


Fig. 13. Microstructure evolution with changing the annealing temperature at deformation temperature of RT (X 50)
a) after annealing at 450°C



b) after annealing at 500°C

c) after annealing at 550°C

5. DISCUSSION

The result of present investigation clearly establishes the effect of the deformation temperature on the recrystallization temperature. A similar effort on 0.43C steel was reported by McQueen and Jonas [13], and the same effort, as above, on polycrystalline copper was reported by Petkovic et al [14,15]. They concluded that, at a higher temperature, more softening was achieved in a fixed time at a same strain, and also recrystallization took place in a shorter time at a same strain. However, since their method of holding after hot deformation was definitely different from the method of present investigation, these results cannot be comparable with the result of present study. They interpreted their results as increased thermal activation of static restoration [13].

Since recrystallization results from microstructural rearrangements toward a recrystallization nucleus, for an interpretation of this result, the study of dislocation density, dislocation arrangement and stored energy is required. In cold deformation, while the flow stress is proportional to the square root of the dislocation density, the distribution of dislocations at high strains may vary from uniform (planar pile ups at low strains) to cellular (entangled). The dislocation arrangement in the higher

stacking fault energy metals at a given strain are more cellular. The influence of the increasing deformation temperature is a sharpening of the voluminous cell walls which will eventually be developed into two dimensional subboundaries, akin to small angle grain boundaries. The effect of increasing flow stress is an increase in dislocation density and a decrease in cell size.

Since driving force for recrystallization is the stored energy and a sharpening of cell walls must represent the lower energy configuration of the dislocations, one takes it for granted that stored energy is decreasing with increasing deformation temperature. However, Fig. 14 illustrates the results, which are plotted as measured enthalpy vs the flow stress in various deformation temperature on <110> copper single crystal [18]. For deformation at 78K, the ideal correspondence, that ΔH is proportional to τ^2, is observed. For the higher deformation temperature, a deviation from this relation is represented in a sence that, with increasing flow stress, the energy increases more strongly than τ^2. For the deformation at 370K, the same tendency is continued. Hence, this result is exactly opposite to expectation. In other words, with increasing deformation temperature, the cell walls become more condensed, but store more energy for the same increment of stress. This does not mean that the higher temperature structure is a higher energy configuration. It

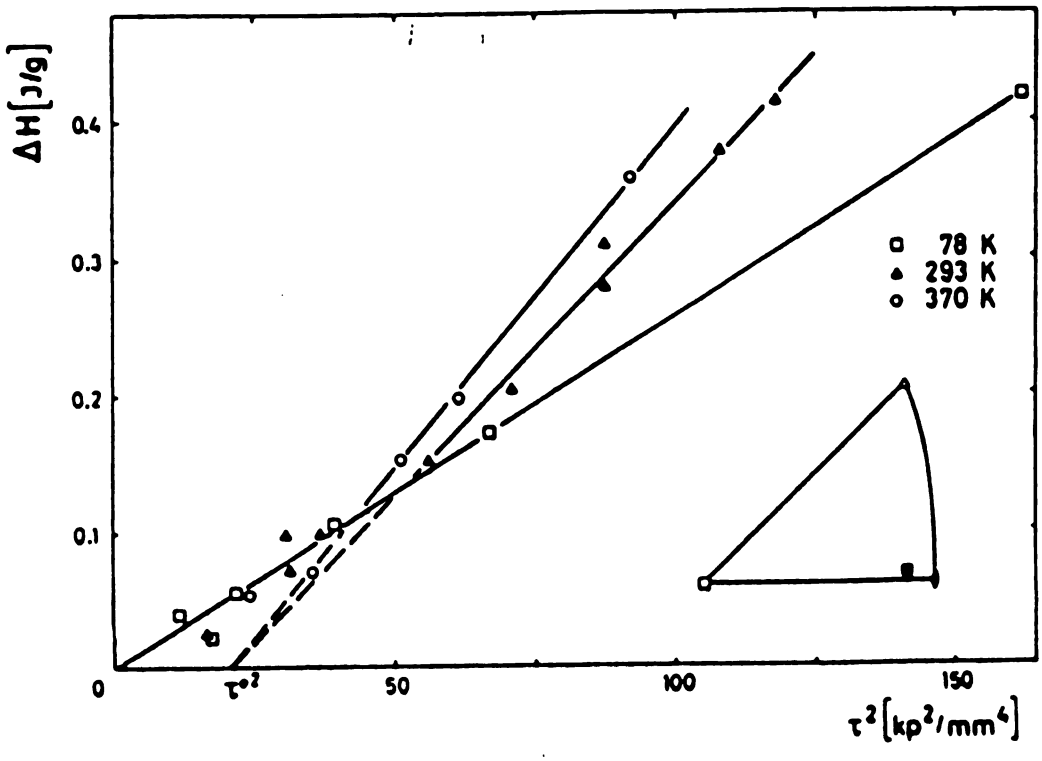


Fig. 14. Measured enthalpy vs flow stress in various deformation temperature on < 110 > copper single crystal.

rather says that the more recovered substructure has a stronger effect to lower the flow stress, which was mentioned by means of Cottrell-Stokes' law previously, rather than the energy. This result indicates that minimum stress for recrystallization decreases with increasing the deformation temperature.

Since the reference stress at each deformation temperature in this study has already been applied to Cottrell-Stokes' law, the present study compares recrystallization temperatures for corresponding deformations at each deformation temperature, rather than the same flow stress. It shows the tendency of a decreasing recrystallization temperature with increasing deformation temperature, even at the same degree of deformation. Since the nucleation of recrystallization is a consequance of locallization of the stored energy, it is possible to interpret this result in a sense that the more recovered structure becomes highly locallized in the stored energy. Therfore, despite of the same degree of deformation, the more recovered the structure, the more liable it is to recrystallization.

6. CONCLUSION

1. In spite of the same degree of deformation, this study shows the tendency of a decreasing recrystallization temperature with increasing deformation temperature. This result indicates that the more recovered the structure is, the more liable it is to recrystallize.

2. Since recrystallization is a consequence of localizing the stored energy, it is possible to interpret this result in a sense that the more recovered structure becomes highly locallized in the stored energy.

3. Due to the concurrent dynamic recovery, the structure becomes locally highly vulnerable to nucleation of recrystallization. This is one key to understand dynamic recrystallization.

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