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Effect of changing load conditions
on Dynamic Recrystallization.

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Sanjeev Deshpande.

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Dr. G.Gottstein
Major professor

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EFFECT OF CHANGING LOAD CONDITIONS ON DYNAMIC RECRYSTALLIZATION

by
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Abstract

Effect of changing load conditions on onset of dynamic recrystallization was studied. Tests were conducted on Cu, single and polycrystal and Ni polycrystal at 0.5 T_m (temperature of melting).

It was observed that recovery plays very important role for the initiation of dynamic recrystallization. In all the tests whenever recovery was enhanced by loading conditions or by static annealing at deformation temperature the dynamic recrystallization (DRX) stress dropped indicating dynamic recrystallization was favored.

In the cyclic tests, cycling in the elastic region showed negligible or very little effect on the recrystallization behavior, but cycling in the plastic region in both directions resulted in dynamic recrystallization at approximately 25% stress level compared with monotonic testing on Ni polycrystal.

All these tests confirmed dynamic recovery is major controlling mechanism for dynamic recrystallization for polycrystals also, similar to single crystals.

Acknowledgements

I take this opportunity to express my sincerest gratitude to my advisor Prof. G. Gottstein, without his help and guidance this project would not have been a success. I would also like to thank my colleague Mark Waterbery for his help in setting up instrumentation, and thanks to all my friends in the MMM Department who helped me in hundred ways sacrificing their time and efforts without any complaints.

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CHAPTER ONE

Introduction

It is established that a variety of f.c.c. materials undergo dynamic recrystallization i.e. recrystallize while being deformed at temperatures above 0.4 T_m, and for the same material at a given temperature and given strain rate the dynamic recrystallization stress is quite reproducible.[1]

This property is very important in all the hot working processes as well for components which are used in high temperature applications.

It is further established that once the critical stress or critical strain (which is approximately 12% for static recrystallization and approximately 22% for dynamic recrystallization while corresponding values for stress are critical stress values) is crossed during deformation eventually the material will undergo recrystallization. This critical strain is approximately 0.8 of the dynamic recrystallization strain also denoted as the peak strain.[2]

Even though these parameters are known, the problem which remains is, it can not be predicted how a change in the deformation conditions affects the occurence of dynamic recrystallization.

Basically two mechanisms can be assumed to control the occurence of dynamic recrystallization.

1] Nucleation

2] Growth of recrystallized grains.

In single crystals there is substantial evidence that nucleation controls the process [3] but in polycrystals dynamic recrystallization may be growth controlled because the dynamic recrystallization stress is much lower. Sakai and Jones have shown that dynamic recrystallization stress keeps on decreasing with each cycle of recrystallization, when starting out with single crystal and deforming through multiple recrystallization cycles.

The obvious reason for this behavior is that grain boundaries in polycrystalline materials affect the dynamic recrystallization such that they provide more favorable nucleation sites in polycrystals which can be activated at lower stress levels. This is not equivalent however, with the conclusion that dynamic recrystallization occurs growth controlled in polycrystals.

In this investigation the problem was addressed by investigating the effect of changes in the strain path on dynamic recrystallization. The strain path of interest, of course, is that prior to the critical strain or the effect of stresses which are less than the critical stress for dynamic or static recrystallization.

Materials selected for the tests were polycrystalline Ni and Cu, and single crystals of Cu. Most of the tests conducted so far are on Cu, because there is much more reference data available for this material and it is easier to grow single crystals of Cu than of Ni. Also the deformation and dynamic recrystallization behavior of Cu is very similar to Ni;[5] this is because the ratio of stacking fault energy to shear modulus and Burger's vector is very much comparable, which controls dynamic recovery and consequently the initiation of dynamic recrystallizaion.

CHAPTER TWO

Brief Review of Previous Results

The occurence of dynamic recrystallization during high temperature deformation is now well established for many metals. Majority of the investigations were conducted in dynamic tests, particularly in torsion, tension and compression.[3, 6]

The dislocation density of a material created during hot working can be reduced by recovery or recrystallization . Both these processes are observed to occur not only under static conditions, i.e. during annealing of cold worked samples, but also under dynamic conditions, i.e. during high temperature deformation.

Once it was established that dynamic recrystallization does occur it has been investigated extensively in polycrystals and the results have proved to be extremly useful in variety of applications, particularly in grain refinement.[3]

But then the physical processes which control the dynamic recrystallization were not understood properly; mainly because of the complex interactions of dislocation tangles, subgrains, high angle grain boundaries, etc. More focus was then given to understand the mechanisms through tests on single crystals in order to simplify the matters by eliminating the grain boundary interactions.

On the Cu single crystal experiments Gottstein et. al. came up with the results that the onset of dynamic recrystallization does not occur randomly during the deformation process. For a constant deformation path, i.e. same material, orientation, strain rate and temperature, dynamic recrystallization is set off reproducibly at a definite value of flow stress. In their experiments they observed the values of shear strain varied on a wide range but the stress remained constant.

So it was concluded that dynamic recrystallization is set off at a critical value of flow stress rather than at a critical strain; this was also observed during present investigation.

In the same paper they came up with results that the actual value of the recrystallization stress depends on the material and on the deformation conditions. With increasing temperature and decreasing strain rate the critical flow stress or the recrystallization stress is observed to decrease. One more important observation during the investigation was dynamic recrystallization in single crystals was found to be triggered by the deformation induced instabilities of the subgrain structure.

The factors which arise this instability can be the controlling factors for dynamic recrystallization.

This kind of study was conducted by Gottstein and Kocks on single crystals of Cu and Ni.[5] From the previous studies on Al single crystals it was observed that it never recrystallizes but always recovers, this strong recovery is due to the high ratio of stacking fault energy to shear modulus and Burger's vector. This particular behavior had led to a hypothesis that dynamic recovery and dynamic recrystallization are two competing processes, but from the experiments on Cu and Ni Gottstein and Kocks came up with a conclusion that dynamic recovery rather than a competing process, is a precondition to occurence of dynamic recrystallization. Dynamic recovery of dislocations leads to rearrangement of cell walls on local scale giving rise to subboundaries[7] which being more mobile can form fluctuations in the homogeneous deformation structure and when subgrains of substantial area are formed can trigger dynamic recrystallization. This conclusion also helped in understanding lower stress values at higher temperatures, where formation of subboundaries is easier and they have higher mobility.

Other than temperature one more factor which is found to be influencing dynamic recrystallization is the strain rate. It was observed that at the same temperature if the deformation is conducted at higher strain rates, recrystallization stress increases.[1]

In the same book they have also shown that by changing deformation path even with same strain rate we get different value of recrystallization stress.

Taking into account all these experimental results we found it of particular interest to find out effect of changes in the deformation path on dynamic recrystallization.

CHAPTER THREE

Experimental Conditions

3.1] Specimen Preparation

Materials - a) Cu Polycrystalline and single crystal 99.99% pure

b) Ni Polycrystalline 99.99% pure

3.11] Mechanical Testing Specimens

All samples for polycrystalline Cu and Ni were machined from 1/2" diameter rods.

The specimen length was 3" with cylindrical gauge section of 1" length and 3/16" diameter.

Cu samples were annealed at temperature of 600 °C for 6 hours, then chemically etched to remove approximately 0.1mm. surface layer with 50% HNO_3. Finally they were chemically polished in a solution of equal parts of H_3PO_4, CH_3COOH and HNO_3 to remove any surface irregularities.

Ni samples were stress relieved at a temperature of 680 °C, then chemically polished to remove approximately 0.1mm layer of the surface with a solution of 70% HNO_3 100ml + 20gms $CuSO_4$.

3.12] Optical Microscopy Specimens

Specimens were cut from the tensile or compression samples by very slow speed diamond wheel cutting along the loading direction and subsequently mounted in cold setting resin before mechanical polishing.

After polishing Cu samples were etched in a solution of 40% HNO_3 + 60% CH_3OH and

Ni samples were etched in 67ml HNO_3 + 33ml CH_3COOH + 1ml HCL.

3.13] Electron Microscopy Specimens

Thin slices of approximately 0.3 to 0.4 mm thickness were cut from tensile samples by slow speed diamond wheel cutting such that the foil plane normal is perpendicular to the direction of loading. These were thinned down to 0.1 mm with 50% HNO_3 + 50% H_2O for Cu and 100ml HNO_3 + 20gms $CuSO_4$ for Ni.

The final jet polishing was done in a Tenupol jet polishing device. Electrolytes used were D2 corrosive (*) for Cu and A8 (*) for Ni.

(*) D2 and A8 are registered trademarks of Struers Scientific Instruments, Inc.

3.2] Mechanical Testing

All tests were conducted on a floor model electro-mechanical Instron testing machine with a 500 kg tension- compression load cell.

Pull rods and button head grips were designed and machined out of 310 heat resistant stainless steel.

To avoid oxidation during testing a cylindrical potective chamber was designed as shown in the fig A. A stainless steel ring with a clearance of 0.75 mm to the pull rod is welded at the top of the tube, while the lower ring which fits on the lower pull rod is made of invar and the dimensions are chosen such that at temperatures in excess of 200 °C a tight fit with the lower pull rod was obtained due to the difference in thermal expansion between invar ring and lower pull rod.

With this chamber designed a protective atmosphere of 90% N_2 + 10%H_2 was maintained during the test inside the chamber to minimize oxidation. Flow rate of 12 litres/hour at 5 PSI was found sufficient to avoid oxidation. Higher flow rates were avoided since gas starts burning at the top of the chamber, also it creates backpressure which should be avoided.

The data aquisition is carried out by A/D conversion by processing the load cell signal into a computer [IBM-XT]. This enabled to store all the load-displacement data from the test at an interval of 1.5 seconds, with an accuracy of greater than 0.1%. This data was then further processed to get the relevant information like, stress-strain curves and workhardening coefficient curves.

Similarly, by the digital output from the computer the movement of the crosshead could be controlled from the computer.

For all the tests strain rate was maintained approximately 2.5×10^{-4} sec^{-1}.

The temperature selected for all the tests was approximately 0.5Tm, so for Cu it was 400 + 5 °C and for Ni 600 +5 °C.

CHAPTER FOUR

Types of Experiments

The experiments can be categorized into the following main groups:

1. Uniaxial tension and compression tests on polycrystalline Cu.
2. a. Tensile deformation to a stress below the critical stress for static or dynamic recrystallization and then holding the specimen for a specific time at the deformation temperature followed by uniaxial tension till dynamic recrystallization.

   b. Similar tests in compression.
3. Deformation of specimens in tension to the same stress value as from 2, removing the specimen, which was then cut to produce the compression samples for:

   a. Compression in the same direction as tension.

   b. Compression at 90° to the tensile direction.
4. Similar tests as in 3 on Cu single crystal.

   a. Tension followed by compression in the same axis.

   b. Tension followed by compression at 90° to the tensile axis but where slip systems remain same.

   c. Tension followed by compression at 90° to the tensile orientation where additional slip systems are activated.
5. Cyclic deformation.

   a. Cyclically loading elastically only in tension at different loads followed by deformation in tension to dynamic recrystallization.

b. Loading cyclically in plastic region in tension and compression with fixed strain amplitude till dynamic recrystallization.

This experiment was conducted on polycrystalline Cu and Ni as well as Cu single crystal.

TABLE I

Test No.	DRX Stress MPa	σ DRX/ σ Saturation	Type of Test
CUTEN1	156.00	0.810	Uniaxial tension
LPCU2	132.00	0.850	Load cycled between 16 Kg. and 76 Kg. in tension. Max. stress 65 MPa.
LPCU5	141.90	0.823	Load cycled between 16 Kg. and 96 Kg. in tension. Max. stress 79 MPa.
LTPCU2	131.65	0.820	Predeformed in tension to 80 MPa. Annealed for 80 minutes at deformation temperature, deformed in tension till dynamic recrystallization.
LTPCU7	126.84	0.850	Predeformed in tension to 80 MPa. Annealed for 20 hrs. Deformed in tension till dynamic recrystallization.
CPCU3	156.00	0.870	Uniaxial compression.

TABLE I (Contd.)

Test No.	DRX Stress MPa	σ DRX/ σ Saturation	Type of Test
LCPCU2	125.80	0.850	Predeformed in compression to 80 MPa. Holding at deformation temperature for 20 hrs. Deform in compression to DRX.
TCPCU2	142.40	0.830	Predeformed in tension to 80 MPa followed by deformation in compression to recrystallization.
TCPCU7	134.30	-	Predeformed in tension to 80 MPa, followed by deformation in compression perpendicular to the tensile axis to recrystallization.
CUPSC3	111.00	-	Strain cycling of Cu polycrystal in tension and compression with strain amplitude of 1.5%.

TABLE I (Contd.)

Test No.	DRX Stress MPa	σDRX/ σ Saturation	Type of Test
NiPSC4	72.00	--	Strain cycling of Ni polycrystal in tension and compression with strain amplitude of 1.0%.
TCSCU5	52.00	0.83	Cu single crystal with <110> orientation for tensile axis, deformed to 25 MPa shear stress in tension, followed by compression in perpendicular direction in <112> orientation.
TCSCU6	55.00	0.86	Cu single crystal, predeformed in tension followed by compression in the same axis.
TCSCU7	38.00	0.86	Cu single crystal, compression axis <111>, perpendicular to the tensile axis <110>.

J

CHAPTER FIVE

Results and Discussions

5.1] Effect of Static Recovery

The hardening curve of Cu polycrystal, deformed in uniaxial tension until dynamic recrystallization, is shown in the figure 1. Dynamic recrystallization is indicated by the maximum stress value on the true stress-strain curve. It was shown before that recrystallization occurs somewhat prior to the peak stress value on the hardening curve, but the difference in the stress values is only marginal so this peak stress will be refered to as recrystallization stress in the following. This is the stress which is shown to be reproducible and the most significant quantity to indicate dynamic recrystallization.

The results are shown in the figure 1. The specimen were predeformed in tension to 80 MPa (approximately 0.5 σ_R), then the test interupted and annealed for 80 minutes and 20 hours. Then deformation was continued until recrystallization occured. From the graphs it is clear that static recovery which is the effect of annealing results in a slight lowering of the recrystallization stress; the 20 hour annealed sample showed 19% decrease in the recrystallization stress. Another interesting point to be noted is, the annealing has also affected the strain hardening behavior. This is indicated by the strain hardening rate ($d\sigma/d\epsilon = \theta$) curves in figure 2. For annealed samples the rate is lower for a given value of stress for a continually deformed specimen. This indicates a change in the subgrain structure. Such a behavior is not observed in polycrystalline Al, Cu or Ni single crystals and is only reported of Al single crystals where it is attributed to subgrain coarsening which is not observed in Cu polycrystal at this temprature. So it is concluded that the presence of grain boundaries affects the dynamic recovery substantially, an important fact for the initation of dynamic recrystallization, which was shown to be recovery controlled.

Similar results were obtained from the samples tested in compression tests.

5.2] The Effect of Strain Path

In this set of experiments the deformation and recrystallization behavior associated with the changes in the strain path were studied.

All specimens were predeformed to 80 MPa (approximately 0.5 σ_R) in tension, followed by compression either along an axis parallel to tensile axis or perpendicular to the tensile axis.

The resulting stress-strain curves are shown in the figure 3. It can be seen that the effect of compression on dynamic recrystallization is very much dependent on the direction of compression axis.

In the tests where compresion is conducted parallel to the tensile axis, there is only slight decrease in the recrystallization stress because same slip systems are activated as per the Taylor's theory, while when the compression is in the perpendicular direction there is substantial decrease in the recrystallization stress. This has to be attributed to the different deformation geometry and so a different development of dislocation substructure. A change in the substructure by operating initially inactive slip systems results in a change in the recovery rate which is indicated by the strain hardening rate curves in figure 4 and therefore a different recrystallization behavior.

In order to substantiate the results from these tests, similar set of tests was conducted on Cu single crystals. The single crystal geometry is shown in the figure 5, where tensile axis was parallel to [110]. Compression was conducted in the axis parallel to tensile axis and along two perpendicular axis [1 1 1] and [1 12], after predeforming the single crystal in tension. The [1 1 1] and [1 12] axes are known to remain stable during compression of f.c.c. crystals. The deformation geometry (figure 5) is such that the compression along [1 12] activated the same slip planes as tension along [1 10], while compression along [1 1 1] activated a slip plane not operated under tension along [1 10]. As can be seen from the figure 6 activation of additional slip plane results in a very strong dynamic recovery and which is reflected by substantial decrease in the recrystallization stress as shown in the figure 7.

5.3] The Critical Deformation Parameters

From previous single crystal experiments [5], it was concluded that the onset of dynamic recrystallization is controlled by the dynamic recovery rate which is reflected by a constant ratio of the resolved recrystalization stress τ_R to the (hypothetical) steady state flow stress: $\tau_R / \tau_S = 0.83$. Since τ and σ differ only by the Schmid factor, also σ_R / σ_S has to be constant. This ratio is plotted in figure 8 as function of recrystallization stress. It is seen that irrespective whether single or polycrystal or whatever the strain history of the specimen, the ratio is invariably 0.84 + 0.03. Hence, the present experiments confirm that dynamic recrystallization is controlled by dynamic recovery in single and polycrystals. Enhanced recovery by changing the strain path, therefore, favors the onset of dynamic recrystallization, i.e. lowers the recrystallization stress.

5.4] Cyclic Deformation

The experiments described in the previous sections confirm that even a prolonged annealing treatment has only a mild effect on the onset of dynamic recrystallization, hence the repeated unloading periods during cyclic deformation should not considerably effect the dynamic recrystallization behavior.

This was also confirmed by a set of tests where cyclic deformation was conducted in tension after monotonic predeformation to 80 MPa (approximately 0.5 σ_R). Cyclic deformation was performed with constant stress amplitude of 65 MPa, i.e. only in the elastic regime, and 79 MPa, i.e. upto the transient to plastic deformation. Cycling was stopped after 150 cycles. Subsequently, deformation was continued by a monotonic tension until dynamic recrystallization occured (figure 9). The effect of cycling in the elastic range is comparable to static annealing for the same period of time without cyclic deformation.

Cyclic tests into the plastic regime were conducted on Ni and Cu polycrystals at 0.5 T_m (i.e. 400 ^{0}C for Cu and 600 ^{0}C for Ni) with constant strain amplitude of 1.5% (for Cu) and 1% (for Ni). The strain rate was same as in the monotonic tests (approximately $10^{-4}s^{-1}$) corresponding to a cycle frequency of approximately of 0.3 or (0.45) min^{-1}. The respective stress-strain curves (figures 10, 11) reveal an initial period of strain hardening followed by a steady decrease of flow stress. The cumulative strain (or number of cycles) to the maximum stress depends on the strain amplitude. For 1.5% strain amplitude (Cu) the peak stress was reached already after 4 cycles, while for 1% amplitude strain (Ni) the peak stress is reached only after 101 cycles, but followed by a more drastic stress decrease in subsequent cycles.

The occurence of dynamic recrystallization was confirmed by metallographic investigations (figures 12, 13). Newly recrystallized grains are observed on the grain boundaries of the deformed material, typical for dynamic recrystallization in polycrystals [9]. The curvature of the boundaries in figure 12 also indicate that strain induced grain boundary migration (SIBM) had occured due to an imbalance of dislocation densities in adjacent grains. Since the volume of newly formed grains is very small, it may be hypothesized, whether SIBM is actually the major softening process under these conditions. Preliminary TEM observations, however, could not confirm this hypothesis.

The dislocation structure in these specimen is a well defined cell structure (figures 14, 15). Compared to monotonic tests up to dynamic recrystallization at the same temperature and strain rate, the structure appears more recovered. The average cell size (linear intercept) is in the order of 5 m, which compares to 1.5 m in monotonic tests at $_R$.

This is reasonable when considering a stress ratio σmon/ σcyc = 300/74 and the commonly observed relationship σ. d = constant. It is remarkable though, that dynamic recrystallization occured in a microstructure with a considerably less stored energy than in monotonically deformed specimens at the onset of dynamic recrystallization. This result substantiates that not the stress but dynamic recovery controls the initation of dynamic recrystallization. The relationship between cyclic hardening, dynamic recovery and microstructure development is presently under investigation by systematically varying the deformation conditions.

CHAPTER SIX

Conclusions

1] The influence of static recovery and the effect of strain path changes on dynamic recrystallization have been studied. Both conditions are relevant to the interpretation of data in cyclic test. It was found that both static recrystallization and load inversion from tension to compression facilitates dynamic recrystallization but only slightly compared to changes to strain paths which change the slip geometry. Low frequency cyclic deformation in tension within the elastic regime has about the same effect on dynamic recrystallization as static recovery for the same period.

2] The initation of dynamic recrystallization is dynamic recovery controlled. Irrespective of the deformation conditions the ratio σ_R / σ_S is approximately constant.

3] Cyclic deformation with a 1% strain amplitude induces dynamic recrystallization after 100 cycles ($T - 0.5\,T_m$, 0.5 cycles/min.) at 25% of the recrystallization stress for monotonic tests under the same deformation conditions. Recrystallization progresses very slowly, maybe assisted by Strain Induced Boundary Migration. The dislocation microstructure corresponds in scale to monotonic tests to the same stress, but the structure appears more recovered.

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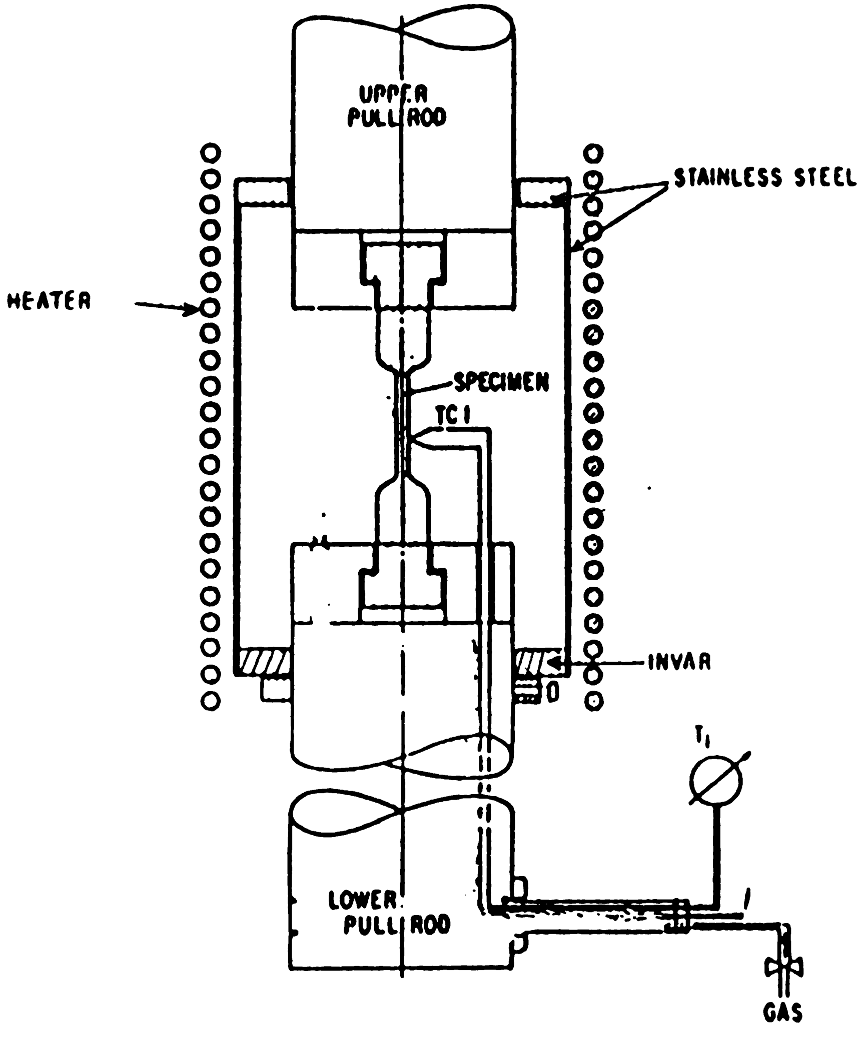

UPPER
PULL ROD
STAINLESS STEEL
HEATER
SPECIMEN
TC1
INVAR
T1
LOWER
PULL ROD
GAS

Figure A

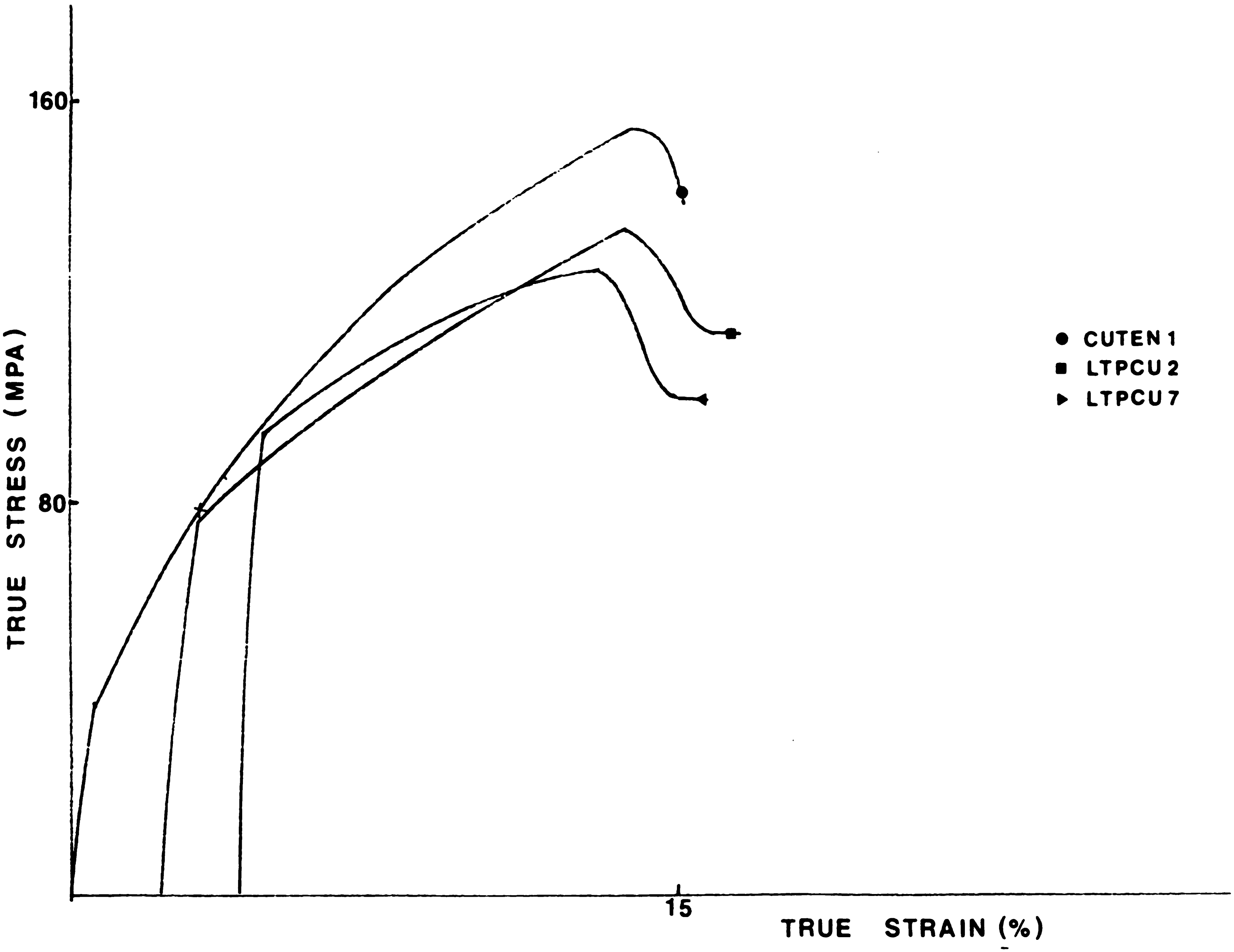


Figure 1

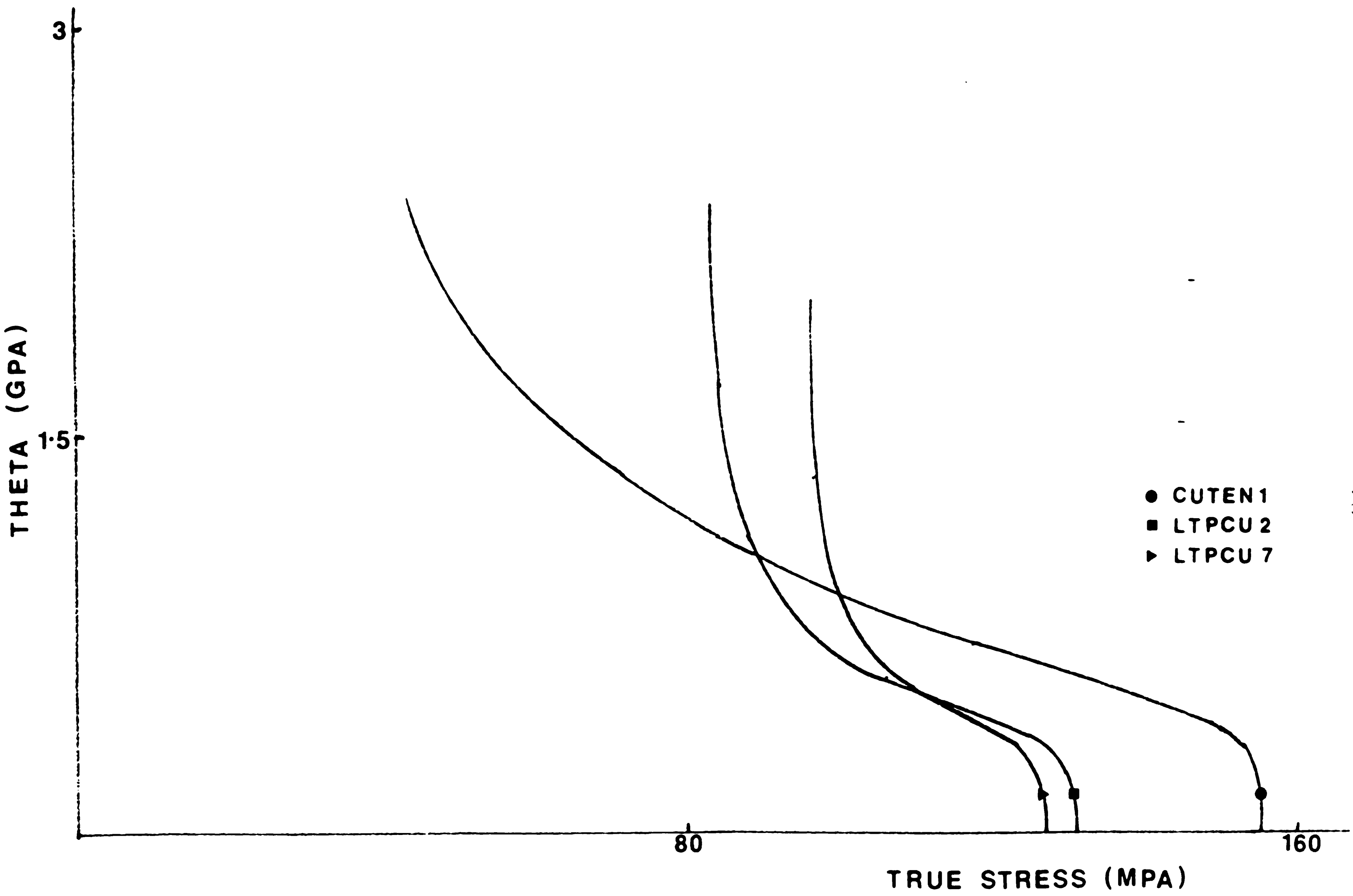
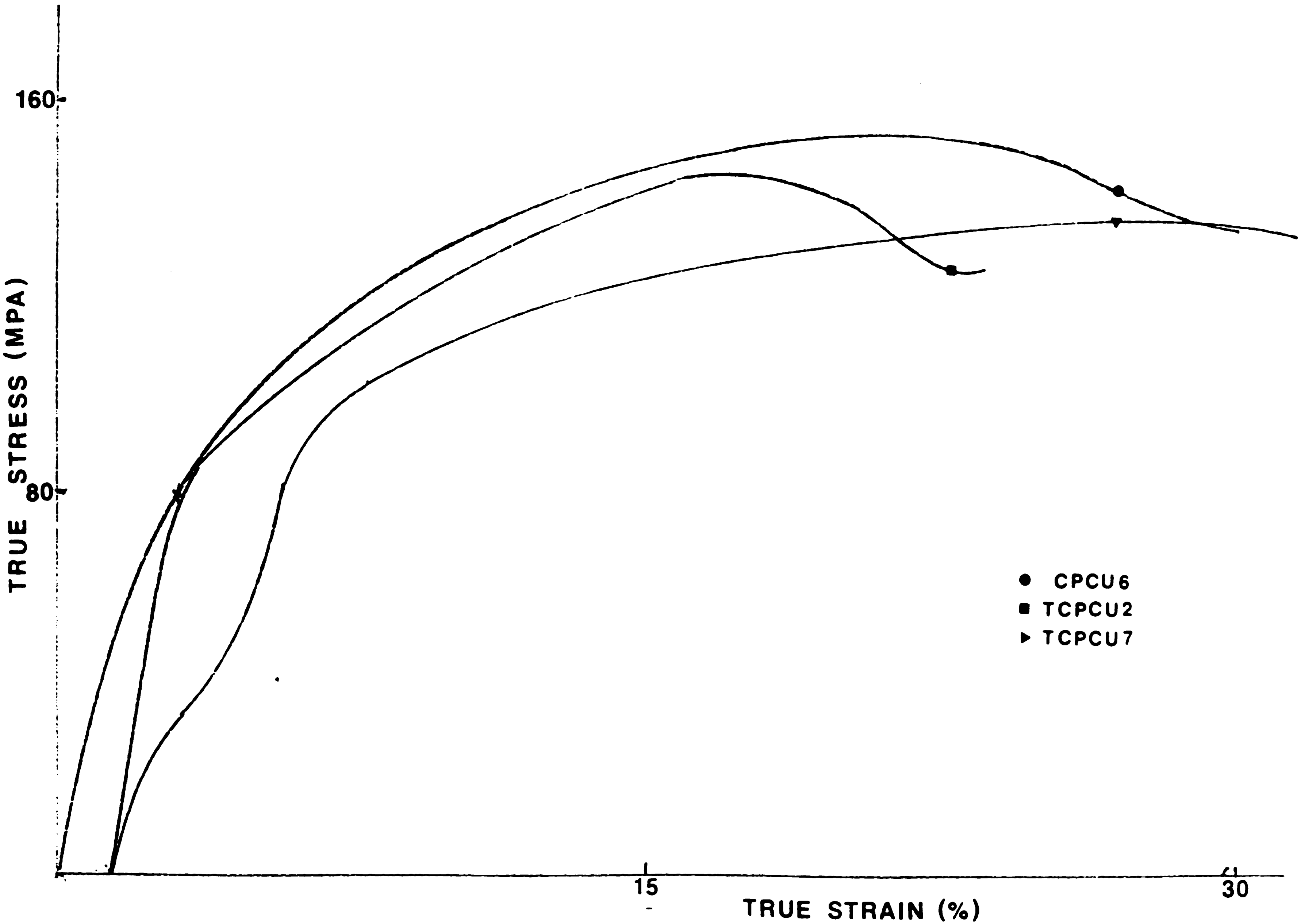


Figure 2



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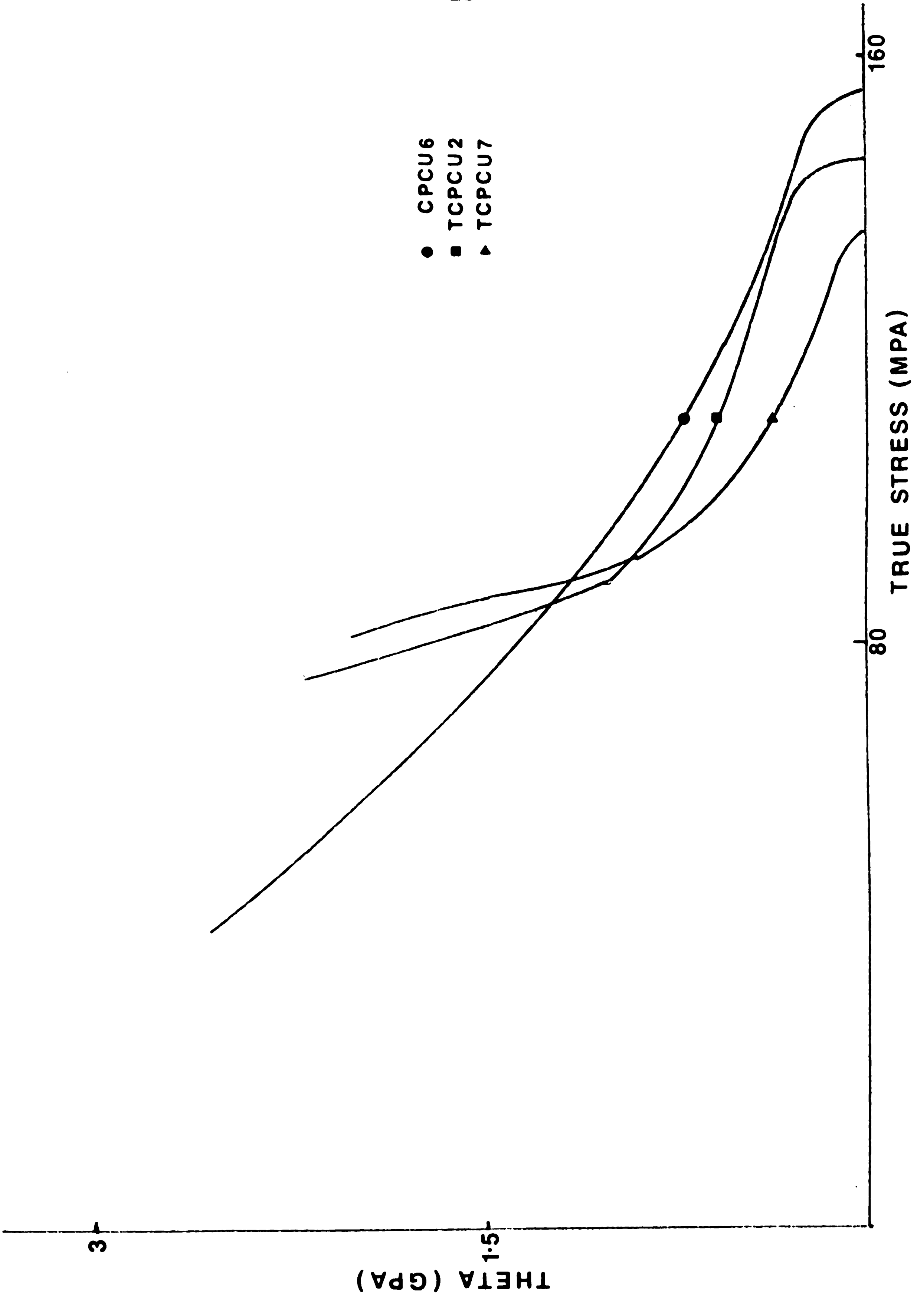

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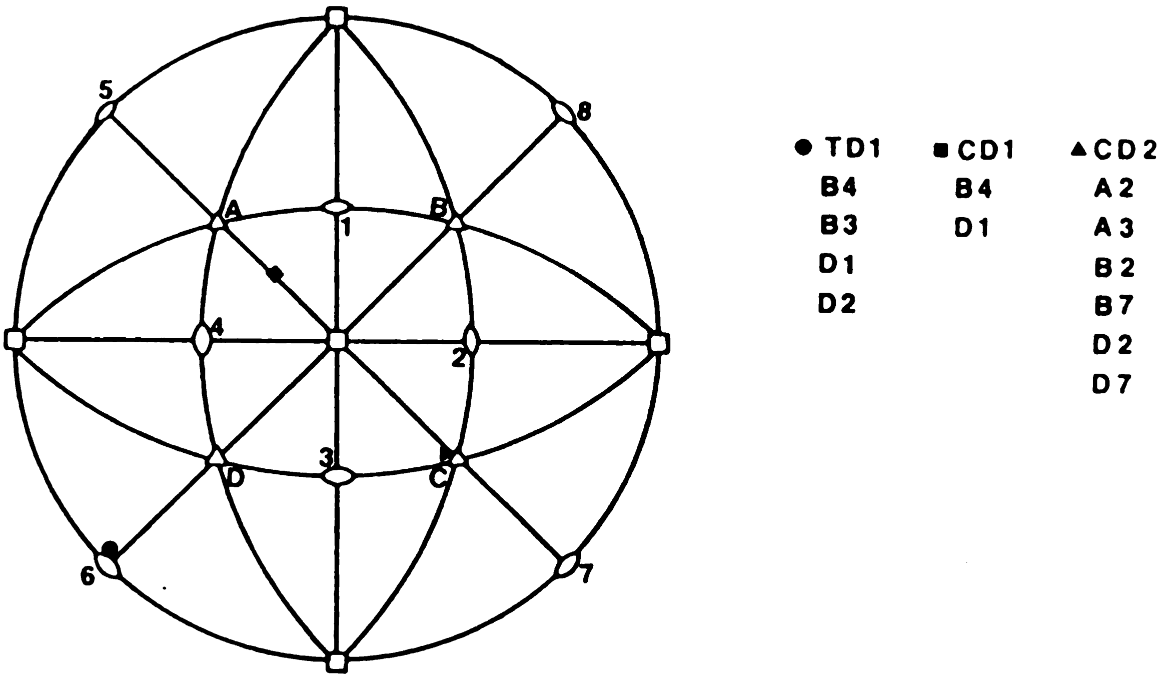

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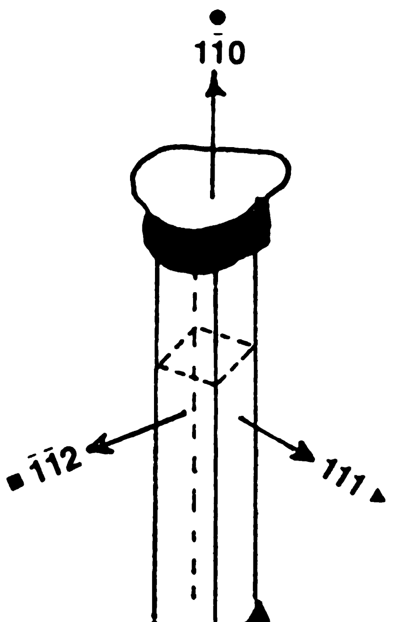


Cu SINGLE CRYSTAL

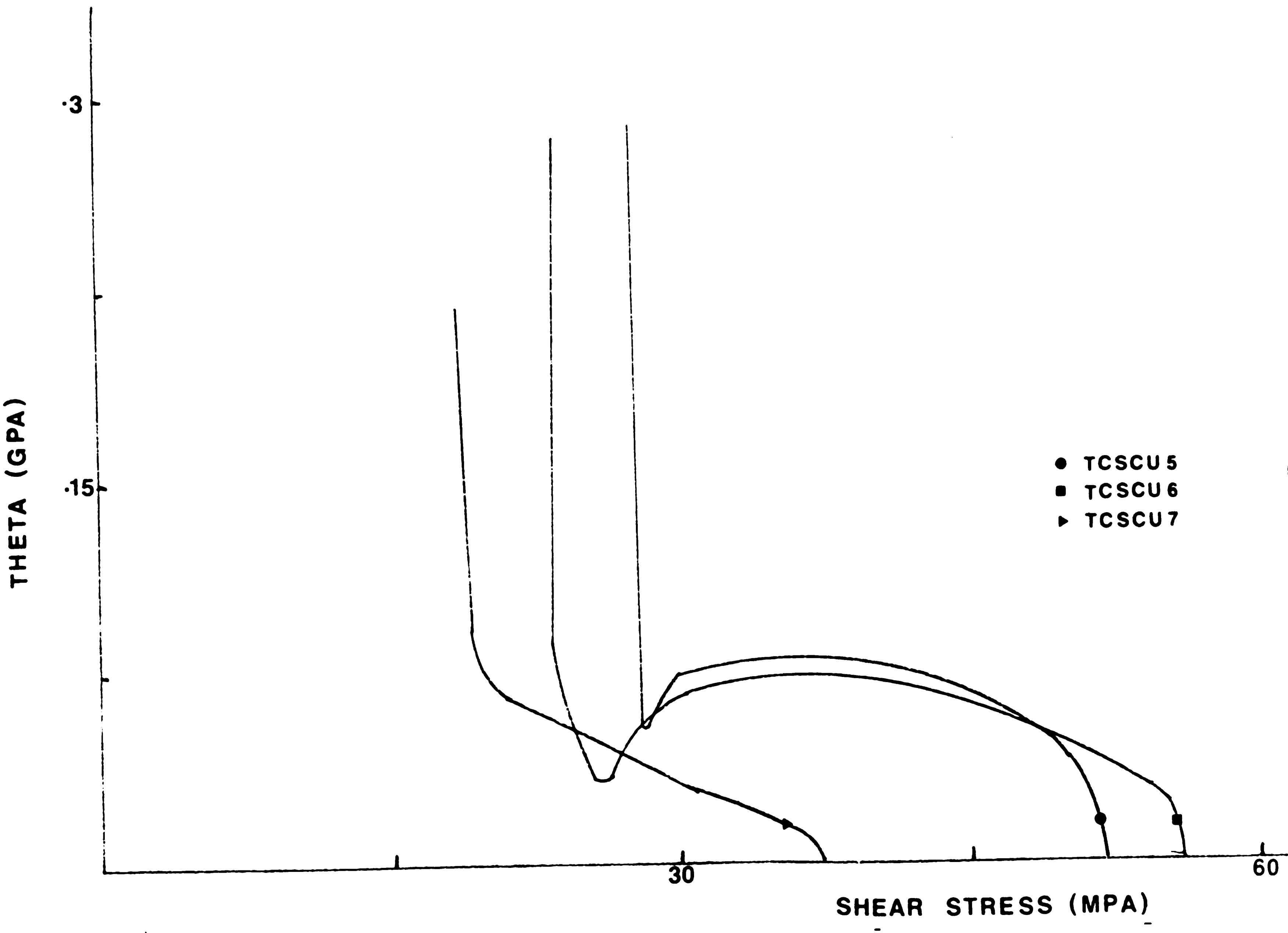


Figure 6

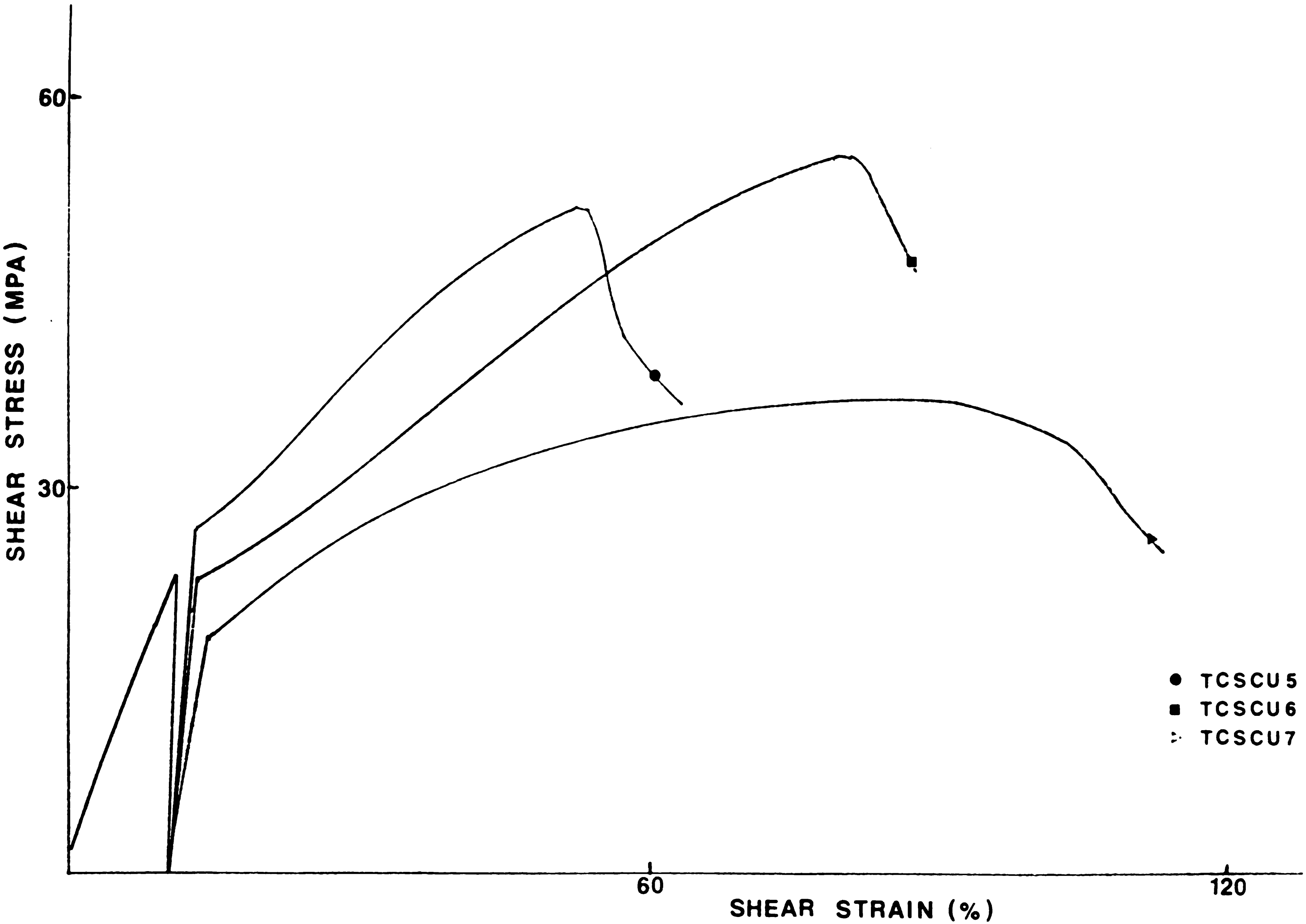

60
30
SHEAR STRESS (MPA)
60
120
SHEAR STRAIN (%)
TCSCU5
TCSCU6
TCSCU7

Figure 7

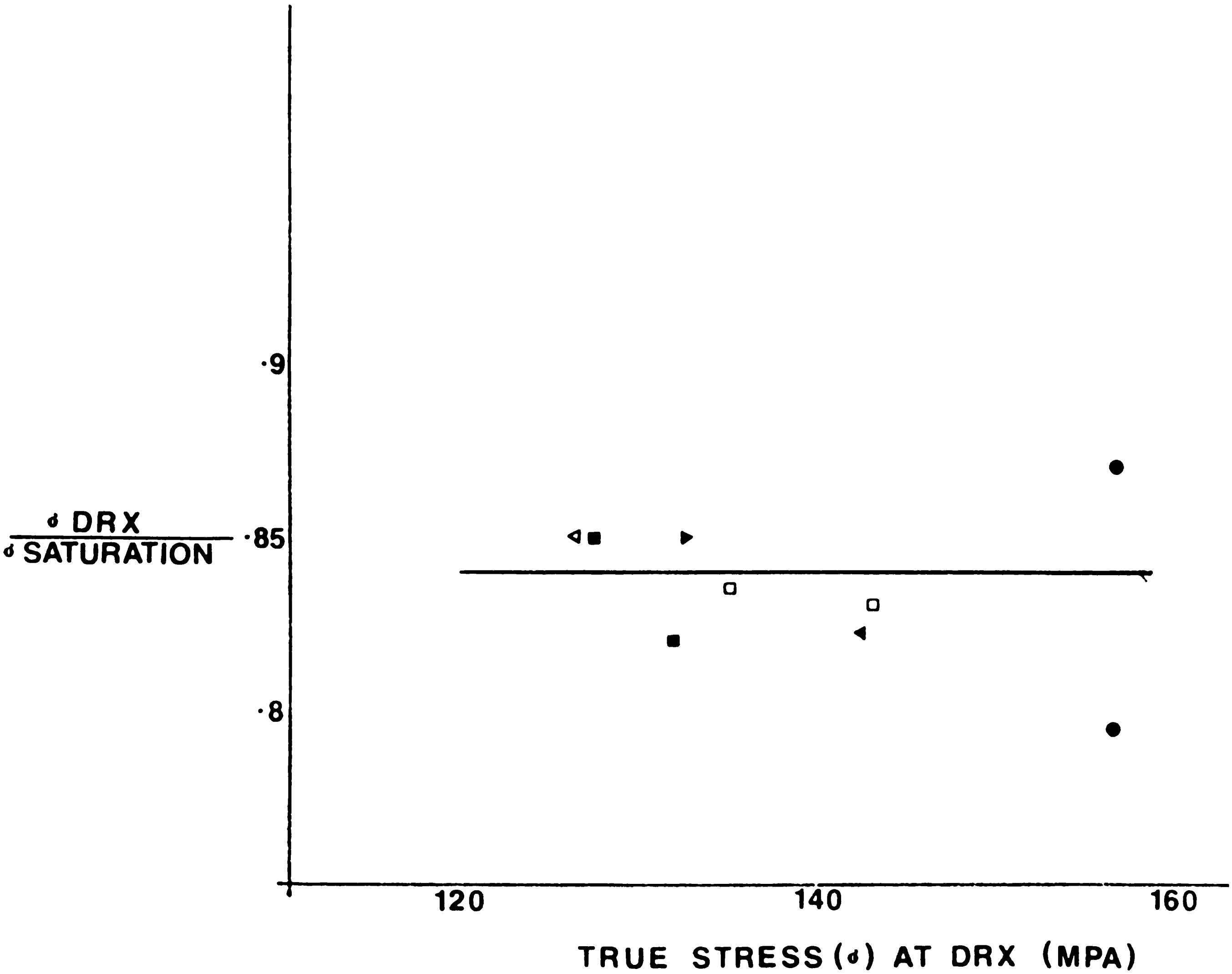


Figure 8

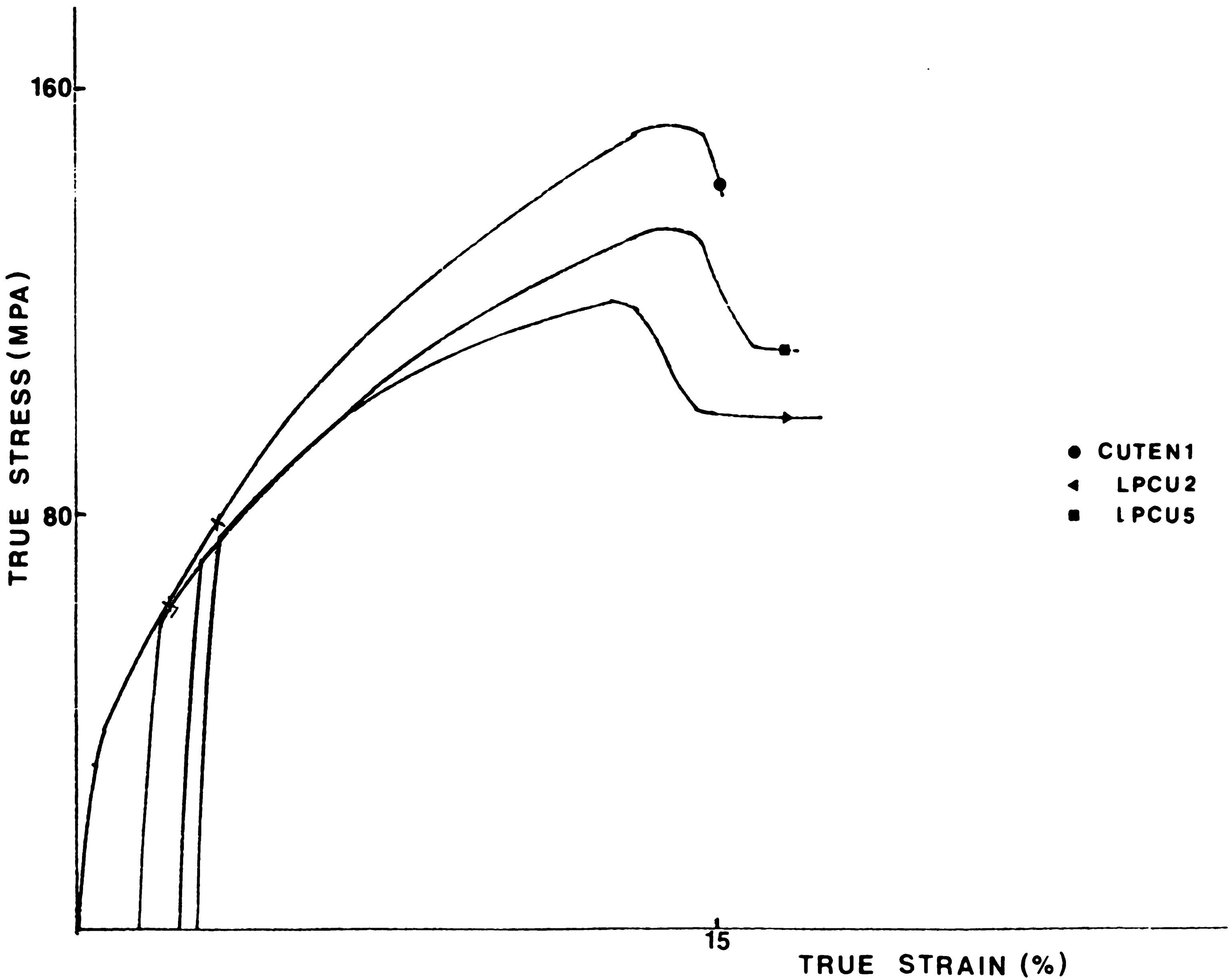
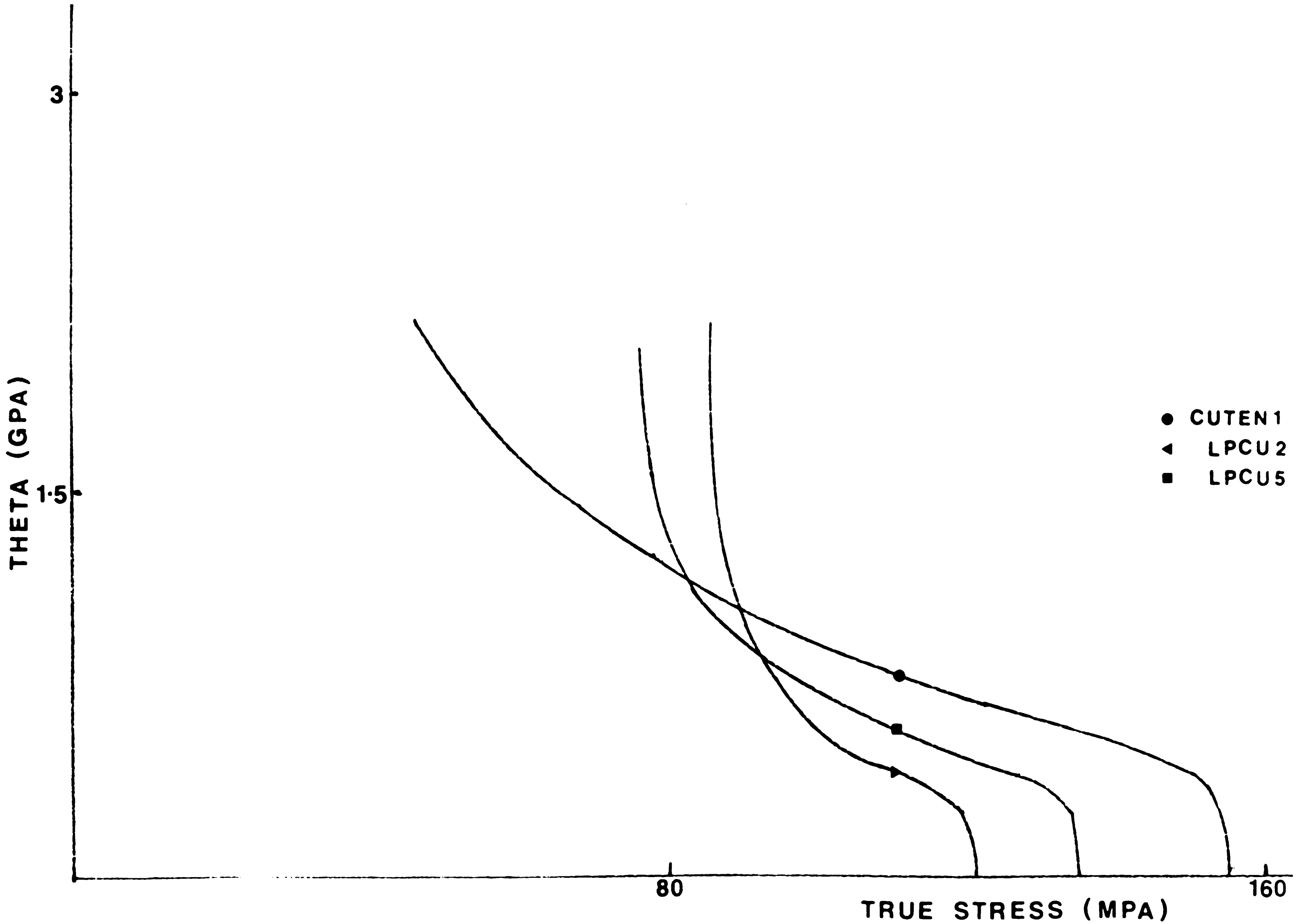


Figure 9(a)



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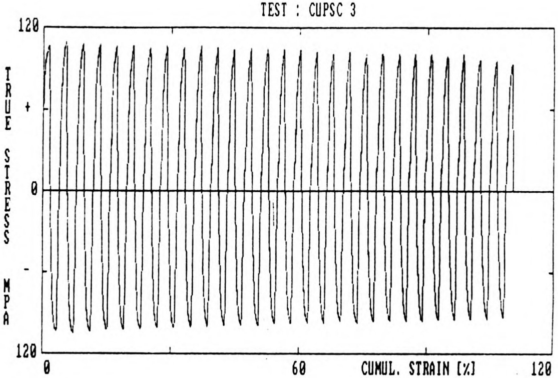


Figure 10

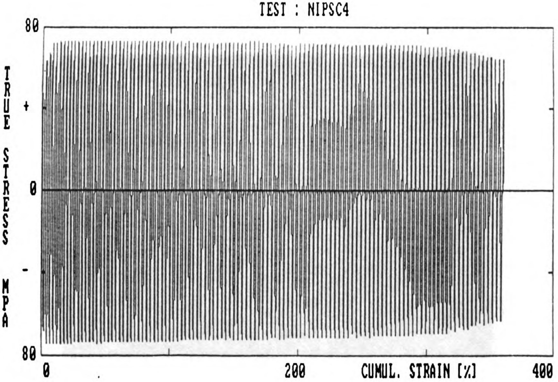


Figure 11

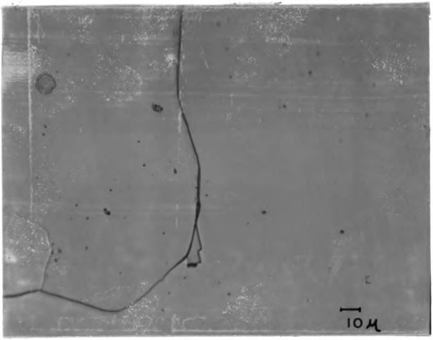
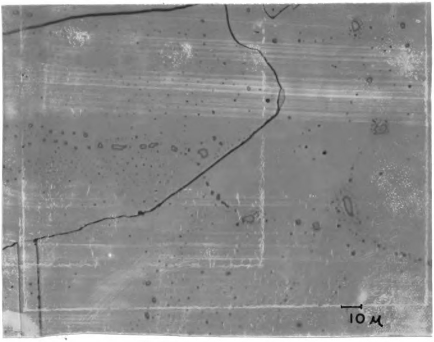


Figure 12



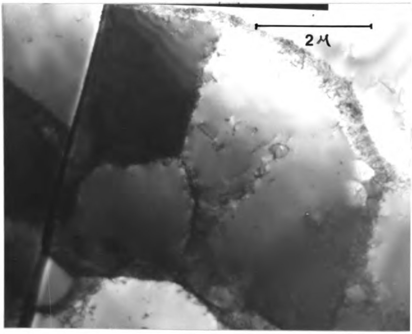
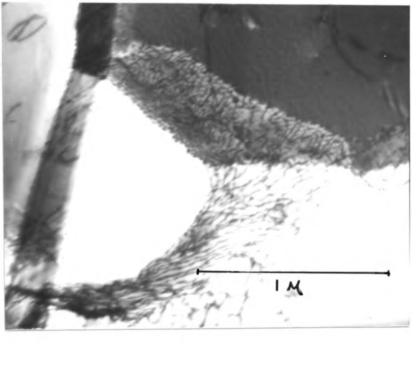


Figure 13 (a)



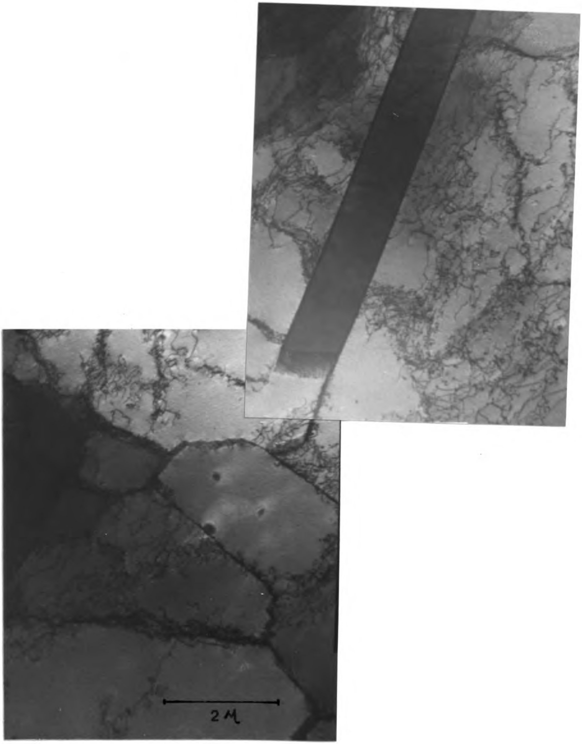


Figure 13 (b)