III-1 Fatigue Fracture of Cu and Cu-Alloy Single Crystals

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Abstract

Single crystals of copper, α - copper-germanium solid solutions, and of Cu_3Au having a single slip orientation were tested in push-pull fatigue at 195° and 300° K. The surface of fatigued specimens was studied by interference microscopy. The deformation behaviour under reversed stress was compared with that under unidirectional stress. The fatigue fracture stress at 10^6 cycles, τ_{10^6}, was related to the thermal cross slip stress τ_{III} and the stress to initiate athermal slip on the cross system in unidirectional deformation. The results are discussed in terms of dislocation mechanisms of fatigue fracture.

1) Introduction

The fatigue fracture of metals at small stresses (relative to the unidirectional fracture stress) is still little understood in terms of atomic mechanisms as compared to other mechanical properties. This is due to the complicated nature of the process itself, and the fact that there are only few observations so far on which a theory can be based. The Wohler or σ/N-curve integrates over too many events including nucleation and growth of cracks during the fatigue life, i.e. number of cycles to fracture N of a specimen cyclically deformed at a stress amplitude σ. It is difficult to locate by surface observations at various stages of fatigue the very few places on the specimen where serious damage is developing. The otherwise powerful tool of electron transmission microscopy is not quite appropriate because of the surface character of fatigue slip: Since it is well known (1-3) that the state of the specimen surface strongly influences the progress of fatigue one would expect that it is near surface layers which show any critical fatigue damage. Only a few transmission observations have so far been reported of such layers (4). Finally, in our experience single crystalline specimens are preferable for the study of processes like cross slip which need a homogeneous critical stress to initiate them. However, the majority of fatigue tests have been carried out on polycrystals.

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It has recently been proposed (1-3,5) that cross slip is important in rationalizing fatigue of crystalline materials at normal or low temperatures. Cross slip is a suitable mechanical softening process known to operate at low temperatures. Also if it is true that a fatigue crack develops steadily during life in a cycled specimen, and that the initial cracks are found in near surface regions, then cross-slipping screw dislocations are the natural means of locally extruding material into the surface (6) in a regenerative manner. It seems difficult to explain by any competing fatigue theory proposed so far (e.g. that of fracture due to condensation of vacancies (7)) that fatigue is strongly surface sensitive and rather insensitive to temperature. These essential observations are, however, in good agreement with the screw dislocation extrusion mechanism operating as follows: the movement of a screw dislocation which lies perpendicular to a free surface creates a surface step, during the first-half strain cycle. This step is removed in the second-half cycle unless slip is transferred to a neighboring plane by means of cross slip. If this happens regularly material will be continuously carried out or moved into a crystal. Crevices or ridges, intrusions or extrusions are formed which will weaken the cross section and finally lead to fracture. This simple mechanism seems to be able to explain most of the basic aspects of fatigue (although perhaps not all aspects of this complex process as a number of authors have rightly pointed out) +).

In order to test such a fatigue mechanism one should change the ability of screw dislocations to cross slip, e.g. by altering the stacking fault energy γ. This is attempted here by alloying copper with a few at% germanium which is well known to lower γ drastically (9, 10). Push-pull fatigue at constant stress amplitude and slip lines are then observed on copper single crystals as a function of Ge concentration and temperature of deformation. Preliminary measurements are also reported on the fatigue of Cu_3Au single crystals tested in different states of order. In an alloy with long range order dislocations must move in pairs which are held together by "anti-phase boundaries" (APB). The energy of such an APB is lower on a $\{100\}$ plane than on the normal $\{111\}$ slip plane. Therefore super-dislocations which are in screw orientation try to cross slip onto a cube plane where they are locked (10a). In a disordered alloy on the other hand cross slip occurs on a $\{111\}$ plane where dislocations can move and contribute to the build-up of the fatigue crack. Fully ordered Cu_3Au crystals therefore should have a higher fatigue strength than disordered crystals.

+) Seeger (8) proposed recently that the interaction between dislocations and clustered vacancies during fatigue deformation leads, as a side effect, to the observed concentration of slip on a relatively small number of "persistent" glide bands in analogy to observations on neutron irradiated crystals.

2. Experimental Details (Cu-Ge)

2.1 Specimen Preparation

Three copper germanium alloys, 1.44, 4.47 and 6.50 at% Ge, were melted in graphite crucibles under vacuum (10^{-4} mm Hg) in an R.F. furnace. Cyclindrical ingots about 1 cm in diameter were obtained by casting into iron moulds, the surface of which had been coated with colloidal graphite. These rods were swaged down to 4 mm. Single crystals were then grown using the Bridgman technique, and the homogeneity was checked by X-ray fluorescence analysis.

By using a seed it was possible to produce all crystals with the same orientation to within $\pm$ 2°. The orientation chosen, favorable for single slip, was similar to that used by Haasen and King (9) so that τ_{III} data of these authors could be used. The single crystals were homogenized for 48 hours at 800-900°C (under 5.10^{-4}mm Hg).

Fatigue specimens were prepared by cutting the single crystals into 4 cm. lengths and hard-soldering cylindrical collars onto the ends. A light electropolish in phosphoric acid was sufficient to remove surface roughness. The final gauge length was 11-12 mm., the diameter 3.8 mm. Fracture on cyclic deformation took place as often in the centre of the specimen as near the grips indicating that the method of gripping did not unduly influence the fatigue mechanism.

2.2 Fatigue Apparatus

The fatigue experiments were carried out in "push-pull" using a PHG 0.4N Schenk-Maschine. The frequency was 2800/minute and the mean stress was zero in most of the tests. A limited number of experiments were conducted at finite mean stresses. Experiments were carried out at room temperature (in air) and at 195°K (in methanol). It was not possible to increase the stress amplitude continuously whilst the machine was running. To avoid starting the test at the full stress amplitude, which causes premature fracture (11), the first 150-200 cycles were applied by hand the stress being increased each 20 or 30 cycles until the desired final stress amplitude was reached. The results are expressed in the form of a Wöhler curve plotting the shear stress amplitude τ against N, the number of cycles to fracture.

2.3 Surface Observations

The surface of the fatigued specimens was studied using optical microscopy and interferometry. Some of them were electropolished after a certain number of cycles, see below. In addition replicas for electron microscopy were taken from specimens deformed in uni-axial tension to the same stresses τ as the fatigued specimens (strain rate $\dot{a} = 1.5 \times 10^{-3}$ sec^{-1}, at room temperature). The deformation of these crystals was interrupted after various strains indicated by arrows in Fig. 1 and replicas obtained from stage I, from the transition of stage I to stage II, and from stage II of the work-hardening curves. The stress chosen for the stage II observations corresponded to the "fatigue limit", τ_{10^6}, given by the Wöhler curves. Replicas were taken from the top surface of the crystal i.e. that part of the surface which the Burgers vector intersects at the largest possible angle.

The slip lines were removed after each replication by electro-polishing before deforming to a higher stress level; the carbon replicas were shadowed at 45° with gold.

3. Experimental Results (Cu-Ge)

3.1 Fatigue under zero mean stress

Figures 2 and 3 show the dependence of the Wöhler curves on concentration and temperature. Each point represents an average of between 3 to 6 single tests. The accuracy of the stress measurement is about ± 2%.

The Wöhler curves as usual show two stages: one - in which we are interested here - of small stresses and large N where the fracture is typical of fatigue failure. This stage is connected to the unidirectional ductile fracture stress (N = ¼) by a transition region of large τ, small N. As is illustrated in Figs. 2 and 3 the addition of Ge to Cu displaces the whole curves to higher stress levels at both test temperatures without altering their shape. Fig. 4 shows the stress required to cause fracture after 10^6 cycles as a function of the germanium content.

The stress τ_{10^6} increases with increasing Ge content, the curve flattening off at 4.5 at% Ge. The curves for 195°K and 300°K diverge, showing that the temperature dependence of fatigue increases on alloying. This behaviour is contrary so that of τ_{III} in unidirectional tensile tests (9).

For pure Cu τ_{10^6} corresponds to the stress for initiation of thermal cross slip τ_{III} as is shown in Fig. 5. τ_{10^6}/τ_{III} at room temperature was about 1.03 and at 195°K about 0.91. Considering that the choice of τ_{10^6} as the "fatigue limit" is somewhat arbitrary and that the τ_{III} data used (12) pertained to different experimental conditions the agreement is sufficient to justify the conclusion that for pure copper the fatigue limit is determined by the onset of dynamic recovery by thermal cross slip +). On alloying τ_{10^6}/τ_{III} decreases however for both temperatures to 0.5 for 6.5 at% Ge. Then the fatigue limit τ_{10^6} for the alloys was better related to the stress τ_{II} in unidirectional strain. Fig. 6 shows that τ_{10^6}/τ_{II} is approximately 2.5 for all concentrations at room temperature and at 195°K, whilst for pure copper this ratio is four to five times larger and differs for the two temperatures.

+) The same result is obtained (3) replotting fatigue limits of polycrystalline pure metals (13) vs. temperature along with τ_{III} data; see also (14).

3.2 Fatigue cycling under non-zero mean stress

A limited number of experiments was carried out on Cu 6.5 at% Ge crystals to investigate the influence of a finite mean stress on fatigue life. Cycling under a mean stress of τ = 2.4 Kg/mm^2, between 0 and 4.8 kg/mm^2, did not cause fracture even after 4.1 x 10^6 cycles. A stress amplitude of 4.7 Kg/mm^2 under zero mean stress causes fracture after 10^5 cycles. Cycling between 0 and 5 Kg/mm^2 also had not caused fracture after 6 x 10^6 cycles.

3.3 Light Microscopic Observation on Fatigued Specimens

Of particular interest were persistent slip bands, extrusions, and intrusions. A pure Cu crystal was cycled at a stress level (±3 Kg/mm^2) designed to cause failure after 10^6 cycles. The test was interrupted after 50,000 cycles and the surface examined. It shows slip lines, some broad, others weak, quite similar to those observed in undirectionally tested specimens. Similar observations have been made by Alden and Backofen (15). Then slip lines were removed by electropolishing and the fatigue test continued at the same stress level for a further 50,000 cycles. Re-examination shows that only a few slip bands continued to be active, about 20 along the gauge length. Interferometric examination at this point revealed the presence of a regular step profile with step heights between 0.03 μ and 0.1 μ. In addition to these steps a few microcracks about 0.1 μ deep and some extrusions were observed (Fig. 7).

Similar investigations on a Cu 6.5% Ge specimen yielded slightly different results. After 50,000 cycles a fine structure was observed which consisted of extrusions. After polishing and a further 50,000 cycles more persistent bands were found than in the case of pure Cu, about 100 along the gauge length. There were no "notch-like" slip steps and the height of the steps was 0.3 μ (Fig.8).

3.4 Electron microscopic observations on tensile specimen

Slip lines were studied at a magnification of about 10^4 after various degrees of tensile deformation at room temperature on Cu 1.5 at% and 4.5 at% Ge alloys.

During stage I in both alloys, the slip lines were long and straight in agreement with similar observations on pure f.c.c. metals (16). Some slip lines on secondary systems were observed during the transition from stage I to stage II.

Further deformation into stage II produced some slip lines which virtually faded out and then continued on a nearby parallel plane, Fig. 9. In a few instances the slip trace linking the two parallel lines was clearly visible, Fig. 10. Whether this trace is visible or not depends mainly upon the shadowing direction. Otherwise the slip line pattern during this stage of deformation is typical of stage II for pure copper.

This linking phenomenon in the alloys can only be explained in terms of cross slip. The stress at which this kind of cross slip occures in the alloys is below that for thermal cross slip, τ_{III}.

Further deformation into the beginning of stage III produced the slip line pattern typical for thermal cross slip, in copper [16, 9].

4. Discussion of the Results (CuGe)

Our experiments reveal a coincidence of the "fatigue limit" τ_{10^6} of pure copper with the stress τ_{III} necessary for thermally activated cross slip in unidirectional deformation at 195°K and at 300°K. This is in agreement with previous results on Cu single crystals [14] and on polycrystalline Cu, Ag, Au [3, 13]. This proves that the activation of thermal cross slip of screw dislocations in pure metals is a necessary and sufficient condition for fatigue softening at large N. For the alloys where τ_{III} is larger (not necessarily because the stacking fault energy is smaller than γ (Cu), see [10]) the fatigue limit τ_{10^6} does not increase proportional to τ_{III} but τ_{10^6} rises rather more slowly with concentration such that τ_{10^6}/τ_{II} is a constant (equal to 2.6). This means [9, 10] that $\tau_{10^6} \approx (4-5)\tau_o$, ($\tau_o$ being the critical resolved shear stress) in the range from 1.5 to 6 at% Ge. For more concentrated polycrystalline α-brasses Robertson et al [17] find that σ_{10^6} even approaches σ_o, the yield stress for 30% Zn in Cu. Our electron microscope slip line observations, although obtained after unidirectional strain, as well as previous observations on α-brass by Maddin et al [18] show, however, that cross slip (in brasses called "prominent" slip on the cross system) becomes visible in the alloys below τ_{III} and in fact just at the stress τ_{10^6}. This type of slip is not observed in pure copper where the slip line structure is generally somewhat finer than in the alloys [9][16]. In agreement with Seeger [19] we interpret this prominent cross slip in the brass type alloys to be athermal slip activity of sources on the cross planes. It appears then that this athermal cross slip in the alloys fulfills the role of thermally activated cross slip in pure copper during the fatigue softening process, i.e. it transfers slip (not necessarily screw dislocations) to parallel planes between two half cycles. Athermal cross slip does not give full dynamic recovery (which in the alloys still needs a stress τ_{III}) as is also shown by an experiment of Avery et al +) (20): An intermediate large twist interrupting cyclic deformation hardens pure copper only for a transition period; but in the Cu - 6% Al alloy, there is a permanent effect. There is (for $\tau \approx \tau_{III}$) dynamic recovery in the former, but not in the latter material as we expected. The fatigue behaviour of the copper alloys thus fits the cross slip model if this is extended to include the(observed) athermal slip on the cross system. The experiments under finite mean stress show that the positive stress half cycle alone does not move dislocations in such a way that they permanently soften the crystal.

+) These authors investigate hardening during the initial stages of bending fatigue on copper and copper-aluminium single and polycrystals. We are not concerned here with the hardening stage and do not follow the conclusions drawn from hardening observations with respect to the fatigue softening mechanism.

Preliminary Results on Cu_3Au

Experimental Details

Single crystals were grown of copper 25 at% gold rods obtained from Degussa, Hanau. The Bridgman technique was used to grow the crystals in graphite crucibles under a vacuum of 6.10^{-5} mm Hg. The as-grown crystals had a diameter of 4 mm which was reduced by spark erosion to [illegible] mm over a gauge length of 6mm with a gradual transition in diameter to the shoulders. The specimens were then electropolished in a hot cyanide solution and annealed for 4 hrs at 830°C in a N_2 - 8%H_2 atmosphere. Some of the crystals were cooled to 500°C and quenched into water, others were slowly cooled in 5 intervals of 2 days each from 365°C to 175°C. The latter treatment according to the x ray lined, resulted in long range order while the former produced a disordered (or short range ordered) structure.

The crystals were fatigued in push-pull using an electrodynamic vibrator (Goodman) at 50 cps and room temperatue. The machine permitted a gradual build-up of the stress over the first 3000 cycles to a preset value. Strain was measured by a linear transformer and was continuously recorded as a function of time. Results on pure copper obtained by this machine were in agreement with those from the Schenk machine, described above.

Results and Discussion

The disordered crystals fractured at a resolved shear stress of [illegible].6 kg/mm^2 after about 2.10^5 cycles. The ordered crystals fractured at 12.6 kg/mm^2 after about the same number of cycles. These stresses appeared to be true fatigue limits. The final crack started along a primary slip trace in the disordered crystals, Fig 11a. In one of the ordered crystals the crack followed a secondary slip trace, i.e. not the surface roughness, Fig 11b. The slip lines on the disordered specimens were coarse and lay along a single set of {III} planes, see [21], while the ordered specimens showed fine multiple slip. Figs.12a and 12b illustrate this as interferographs. The regularity of the surface roughness due to fatigue slip is of different scale depending on order.

Neither x-ray asterism nor a change in orientation nor a change in the state of order were observed for any specimen.

The fatigue limits $\tau^{d/o}_{2.10^5}$ are to be compared with the critical shear stresses in tension of fully ordered, τ^{o}_{o}, and disordered, τ^{d}_{o}, Cu_3-Au single crystals, respectively. According to Kear (10a) $\tau^{d}_{o} \approx 4 kg/mm^2$, τ^{o}_{o} = 1.4 kg/mm^2 at 300°K. Therefore $\tau^{d}_{2.10^5}/\tau^{d}_{o} = 2.55$, $\tau^{o}_{2.10^5}/\tau^{o}_{o} \approx 9$. For the disordered alloy, $\tau^{d}_{II} \approx \tau^{d}_{o}$ and the above ratio is found to be almost identical with that measured on the CuGe alloys. (See § 3.1). A shear stress of 10.6 kg/mm^2 lies somewhere in stage II of the unidirectional stress strain curve. τ^{d}_{III} is not known due to the interference of conjugate Lüders strain. Since nonthermal cross slip on {111} planes is reported (10a) after deformation of disordered Cu_3Au into the beginning of stage II we assume the mechanism postulated in § 4 to operate also in this alloy. The stacking fault energy γ^d of the disordered alloy is not known but it is probably as low as for its components (10). From x-ray line shifts observed on Cu_3Au filings Mikkola and Cohen (22) estimate $\gamma^o < \gamma^d$. The strong tendency towards short range order in disordered Cu_3Au will help to concentrate slip onto a few planes in agreement with observations by transmission electron microscopy (10a).

The high fatigue strength $\tau^{o}_{2.10^5}$ of long range ordered Cu_3Au relative to τ^{o}_{o} is at first surprising, especially in any fatigue model that depends on intersecting slip, although the ratio of these stresses is only half that found for pure copper. The stress τ^{o}_{II} cannot be measured because there is very little easy glide here. τ^{o}_{III} for ordered Cu_3Au on the other hand is enormously high, greater than $6.\tau_{III}(Cu)$, and is not reached before fracture at 300°K (10a). Thus thermal cross slip of the super-dislocations is impossible. Athermal cross slip in ordered Cu_3Au should be seriously hindered relative to the disordered alloy by the self-locking of screw super-dislocations on cube planes (10a) as pointed out in §1. This will be studied further.

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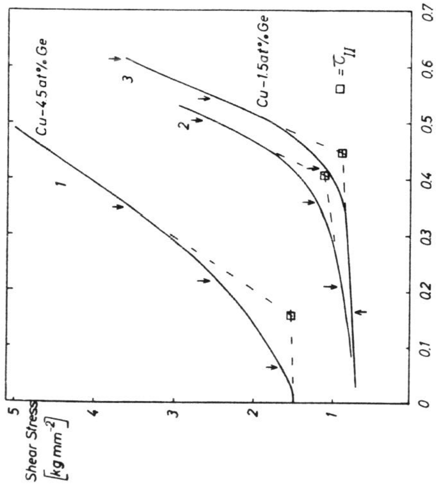


Fig.1 stress strain curves of Cu-Ge single crystals at room temperature in tension. Positions of replica observations marked by arrows.

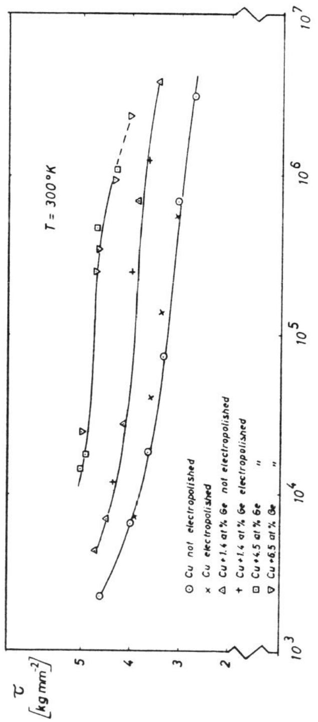


Fig.2 Wöhler curves of single crystals at room temperature.

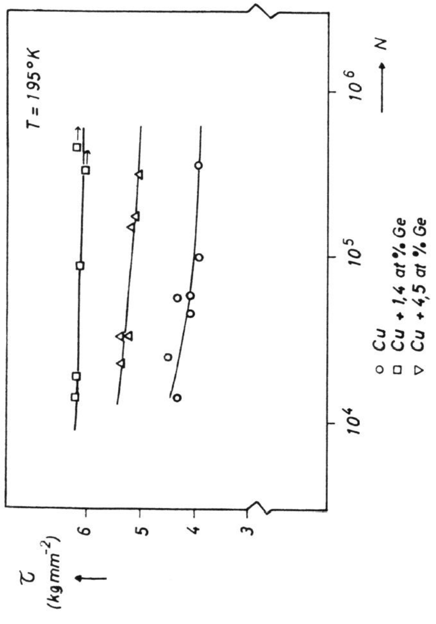


Fig.3 as fig.2, only at 195°K.

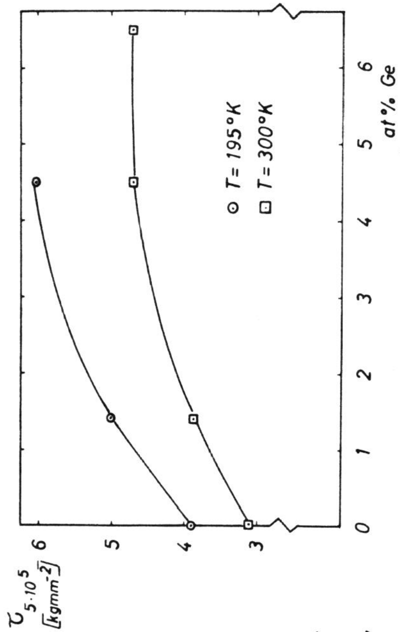


Fig.4 Fatigue limit $\tau_{5\cdot10^5}$ vs. Ge concentration at two temperatures.

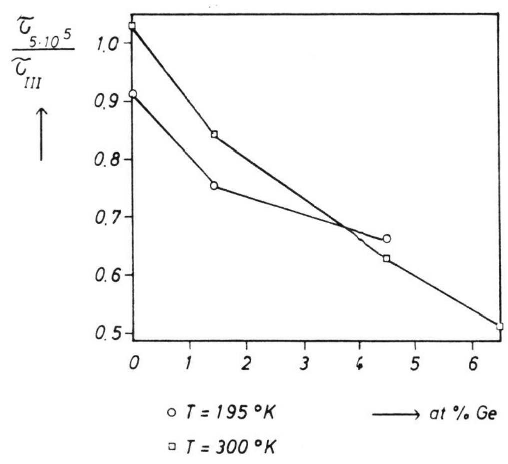


Fig.5 Ratio of fatigue limit to unidirectional thermal cross slip stress vs. Ge concentration.

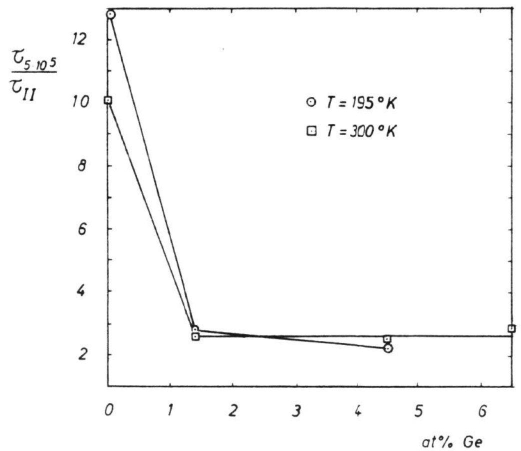


Fig.6 Ratio of fatigue limit to initiation stress τ_{II} for secondary slip in tension.

Fig.7 Interference micrograph of copper crystal fatigued to 5.10^4 cycles at τ = 3 kg mm^{-2}, repolished, and cycled for another 10^5 cycles. Magn. 450x.

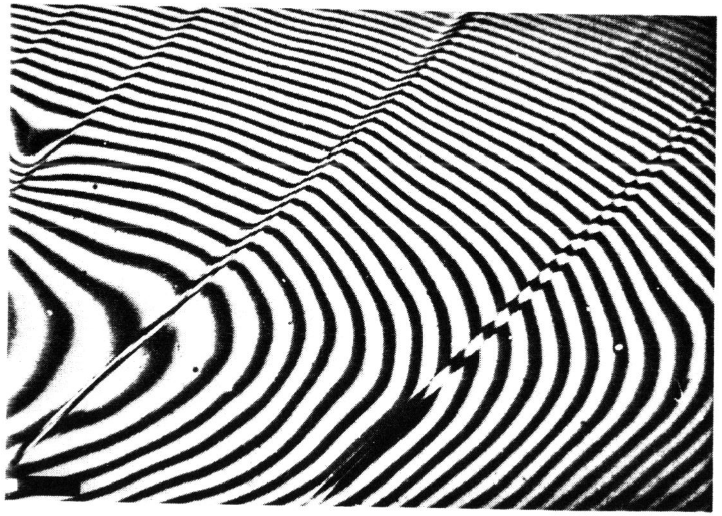


Fig.8 as fig.7, only for Cu 6.5% Ge crystal, cycled at τ = 4.7 kg mm^{-2}.

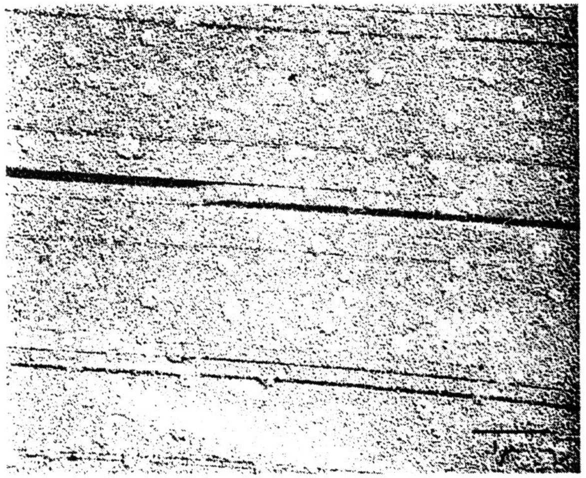


Fig.9 Electron micrograph showing athermal cross slip in stage II at τ = 3,56 kg mm^{-2}, for Cu 4.5% Ge alloy (curve 1, fig.1) magn. 15.000 x.

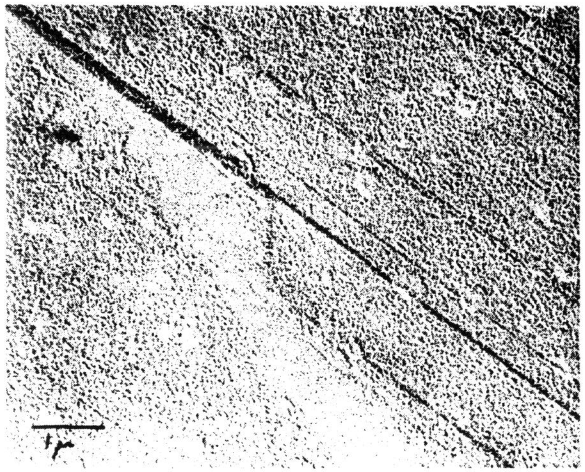


Fig.10 As fig. 9, magn. 25.000 x.

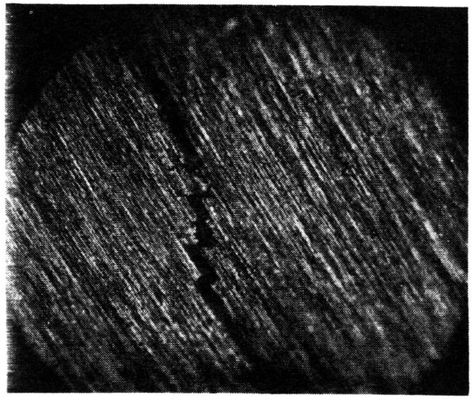


Fig.11a. Crack parallel to main slip planes in disordered Cu_3Au after 2.10^5 cycles, Magn 240 x.

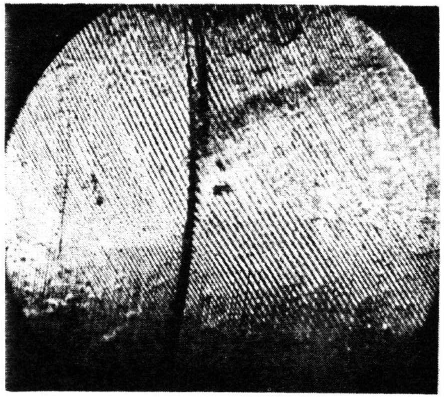
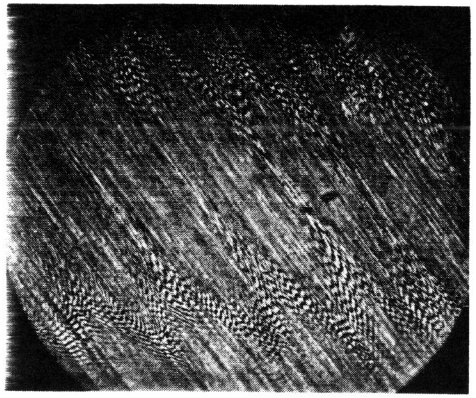


Fig.11b. Crack following secondary slip trace in ordered Cu_3Au, 2.10^5 cycles, magn 96 x.



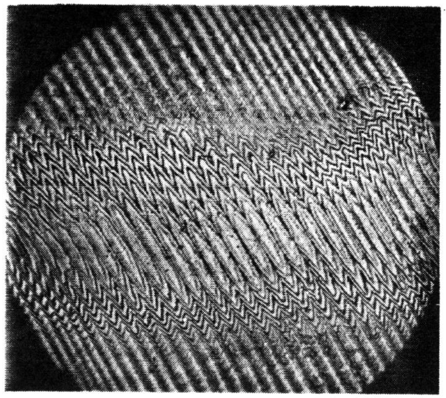


Fig.12a. and 12b. Interferographs of disordered and ordered Cu_3Au, respectively, after 2.10^5 cycles, magn 230 x.