DEPARTMENT OF ENERGY, MINES AND RESOURCES. MINES BRANCH OTTAWA

CRITICAL RESOLVED SHEAR STRESS OF LEAD

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PHYSICAL METALLURGY DIVISION

Reprinted from the Canadian Journal of Physics.

Volume 45 (1967)

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ROGER DUHAMEL, F.R.S.C.

Queen's Printer and Controller of Stationery

Ottawa, Canada

1968

CRITICAL RESOLVED SHEAR STRESS OF LEAD!

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Measurements of the critical resolved shear stress of lead are reported as a function of temperature, purity, solute additions, and orientation. By annealing in situ it was found that the value of the CRSS can be reduced, the scatter between specimens decreased, and the same specimen can be tested a number of times.

Over the temperature range 4.2 °K to 600 °K (the melting point) the CRSS decreased from 53 g/mm² to approximately 10 g/mm². Between 100 °K and 300 °K the temperature dependence of the CRSS is the same as that of the shear modulus.

It was found that the CRSS is relatively insensitive to differences in the trace impurity level and to solute additions of 0.1% Sn and 0.02% Cu. Additions

of 1.0% Sn appreciably increase the CRSS at low temperatures.

The orientation dependence of the CRSS is similar to that shown for copper, with higher values at the edges and corners of the stereographic triangle.

Under optimum conditions the average value of the CRSS of lead is 34 g/mm^2 at 78 °K. This value is anomalously high when compared to that of copper, using $\sigma \propto b\mu N^3$ for the flow stress to make the comparison.

INTRODUCTION

The critical resolved shear stress of metal single crystals is generally considered to be markedly sensitive to trace amounts of impurities, and to the detailed procedures of growing, handling, and testing specimens. Wide scatter is observed for apparently similar specimens. As a result it is difficult to find reliable data in the literature for the CRSS of pure metals and, in particular, the temperature dependence of the CRSS.

The CRSS of aluminium has been examined extensively by Lucke and Buhler (1963) as a function of temperature and strain rate. They obtained highly consistent values for the CRSS, and determined its temperature dependence over the range 300 to 900 °K. They compared their results with the temperature dependence of the flow stress for aluminium as determined by Hirsch and Warrington (1961) and obtained good agreement. They also calculated the activation energy of the deformation process for the high-temperature region, using their values of the temperature and strain-rate dependence of the CRSS, and obtained a value of 1.36 eV, which agreed very well with the activation energy of self-diffusion of aluminium, 1.35 eV. These results provided confirmation of the Hirsch and Warrington theory of high-temperature deformation, which proposed that the rate-controlling process was the rate of movement of vacancies from dislocation jogs and was the same as that of self-diffusion. Hirsch and Warrington's determination of the activation energy, using flowstress-ratio and strain-rate-sensitivity measurements, gave a value of 1.6 eV, somewhat higher than that of self-diffusion.

The general expression for the flow stress of an f.c.c. single crystal is given by (see Nabarro, Basinski, and Holt 1964)

¹Presented at an international conference on the Deformation of Crystalline Solids, held in Ottawa, August 22–26, 1966.

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(1)
$$\sigma = b\mu N^{\frac{1}{2}}/\omega,$$

where σ is the flow stress, b is the Burgers vector, μ is the shear modulus in the operative slip plane and slip direction, N is the dislocation density, and ω is a constant having a value between 3 and 5. Assuming that this expression for the flow stress is applicable to the CRSS, and that there is relatively little difference in the value of N for f.c.c. crystals grown under normal conditions in graphite, then the CRSS of, say, lead and copper should be proportional to $b\mu$.

The values of b for lead and copper are 3.49 and 2.55 Å, and those of μ are 0.73 and 4.08 \times 10¹¹ dynes/cm² respectively, indicating that σ_{Pb}/σ_{Cu} should be 0.25. The average value of the CRSS of copper at 78 °K is of the order of 40 g/mm² (Basinski 1966), which would predict a value for lead of 10 g/mm². The value for zone-refined lead, as reported in the literature, is 55 g/mm² (Bolling et al. 1962), which is much higher than that predicted.

The purpose of the present investigation of the CRSS of lead was to determine whether reliable and reproducible data of the CRSS could be obtained. If this could be done, then the temperature dependence would be determined and the high-temperature deformation process analyzed in a manner similar to that of Lucke and Buhler. In addition, the effect of orientation, impurity level, solute hardening, and prior deformation on the CRSS would be determined. From these data the minimum value of the CRSS could be compared to that of copper as considered above.

EXPERIMENTAL PROCEDURE

The material used in this investigation was 99.999% (59) and 99.9999% (69) purity lead provided by the Consolidated Mining and Smelting Company.

Crystals were grown in a horizontal graphite boat in the form of a plate, using graphite inserts and a cover. The test specimens were rectangular in section (0.65 \times 0.33 cm) and had a 5-cm gauge length. At each end of the gauge length the crystal widened from 0.65 to 1.3 cm with a 45° taper on either side. To grow the crystals, pieces of "as-received" metal were placed in one end of the boat, melted in a vacuum of 5 \times 10⁻⁵ Torr and allowed to flow under the cover, through an orifice, into the region where the test specimen was grown. Specimens prepared from 59 lead or low-concentration alloys were generally seeded on to oriented crystals. With 69 lead seeded crystals could not be grown successfully; however, there was no difficulty in growing unseeded crystals which were subsequently used for testing.

After growth the ends of the specimens were carefully removed with a jeweller's saw, at points well away from the gauge length, and the specimens tested without etching or polishing to keep deformation due to handling to a minimum.

The sections cut from the specimen were used to determine the crystal orientation, using standard Laue back-reflection techniques. All of the specimens exhibited a lineage structure which was generally highly irregular and varied with the orientation of the specimen. The specimens were grown at 8 cm/hour.

In some cases the pure lead specimens were annealed in a silicone oil bath prior to testing, as were all alloy crystals, for the time and temperature indicated. In general the specimens were annealed *in situ* in the tensile machine using a well-stirred silicone oil bath at 285 °C for 20 minutes.

A number of specimens of pure lead were analyzed after testing, to determine whether impurities were picked up in the growing and handling process. The analysis was done by the Consolidated Mining and Smelting Company. It was found that some of the specimens had picked up as much as 30 p.p.m. Cu and 20 p.p.m. Zn, which was attributed to heating one of the brass end plates of the vycor tube containing the graphite boat. The brass ends were subsequently water cooled, and analysis of the lead specimens after testing showed no pickup. This analysis for 69 lead was 0.5 p.p.m. Cu, <0.1 p.p.m. Mg, 1.0 p.p.m. Si, and <0.1 p.p.m. Ag. An oxygen analysis was also made and the oxygen content found to be 4 p.p.m. The presence of the 30 p.p.m. Cu and 20 p.p.m. Zn had no observable effect on the CRSS.

The lead crystals were deformed in a table model Instron fitted with a yoke, to enable the specimen and supporting rods to be fully immersed in a silicone oil or low-temperature bath. Small steel grips were used to hold the specimen, such that the stress was applied along the 45° flange at the end of the specimen by suitable flanges in the grip. Since the specimen was alternately immersed in hot silicone oil and liquid nitrogen, it was impossible to use ball joints or flexible couplings to take up the expansion and contraction of the specimen and supporting rods with change in temperature. The specimen grips were rigidly clamped in position and the cross-head position manually adjusted when the temperature was changed, keeping the stress to within 0.2 of the onset of flow. The rate of strain was normally $1.7 \times 10^{-4} {\rm sec}^{-1}$.

The CRSS was determined from the load-elongation curves by extrapolating both the elastic and plastic (stage I) portions of the curve to their point of intersection. This was normally clearly defined in the single-glide specimens and did not differ significantly from the point delineating the onset of plastic flow. The $\langle 100 \rangle$ and double-glide orientations exhibited parabolic hardening at low temperatures and the CRSS was not as clearly defined in these cases. The value of the CRSS was determined from the flow stress using the Schmid factor and specimen diameter.

The alloy crystals were grown in the same manner as the pure lead crystals, the low alloy concentrations having controlled orientations and the high alloy materials random orientations. "As-grown" samples of each run were chemically analyzed to ensure that there was no macrosegregation in the gauge length and to determine the correct "as-grown" composition. In general the alloy crystals were annealed for 80 hours at 550 °K in silicone oil for homogenization prior to testing.

OBSERVATIONS

The results of the first series of measurements of the CRSS of both 59 and 69 lead single crystals oriented for single glide are shown in Fig. 1. Each point corresponds to a separate specimen. The temperature dependence of the shear modulus is also shown, as obtained from published data (Waldorf 1962) up to 300 °K and extrapolated above this temperature.

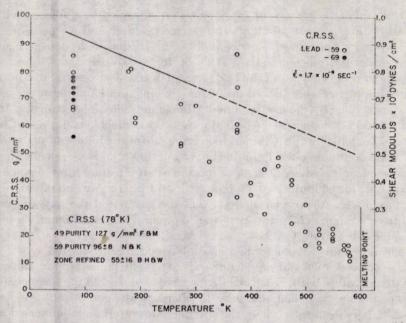


Fig. 1. Critical resolved shear stress of lead single crystals, oriented for easy glide, as a function of temperature. The temperature dependence of the shear modulus is shown by the solid line. Published values of the CRSS at 78 °K are given at the lower left.

It is apparent in Fig. 1 that there is a wide scatter in the values of the CRSS at any given temperature, this scatter progressively decreasing with increasing temperature. At 78 °K, where the results for 59 and 69 lead are shown, there appears to be no significant difference between these two materials. The average value of the CRSS at 78 °K is 72 g/mm², which is higher than the value of 55 g/mm² reported by Bolling *et al.* (1962) for zone-refined lead and lower than both the 96 g/mm² for 59 lead (Neureth and Koehler 1951) and 127 g/mm² for 49 lead (Feltham and Meakin 1957) as might be expected. At increasing temperatures the CRSS decreases approximately proportional to the modulus. However, the scatter is much too wide to make any significant comparison.

It was found that the scatter of the CRSS could be reduced appreciably and the same specimen tested a number of times by annealing the specimen *in situ*. Annealing specimens before mounting in the Instron did not make any appreciable difference in the scatter of the CRSS.

The effect of annealing *in situ* on one specimen is shown in Fig. 2. The specimen was mounted in the Instron, annealed for 30 minutes in silicone oil at 550 °K, cooled to 78 °K, pulled 0.1% to determine the CRSS, reannealed, and pulled again for a total of eight cycles. The average CRSS is 44 g/mm² as compared to the average of 72 g/mm² at 78 °K for the unannealed specimens shown in Fig. 1, and the hardening due to the 0.1% strain is fully recovered by the annealing process.

Fleischer (1961) has pointed out that since lead recrystallizes at room temperature, strains in this material should anneal out at room temperature. This

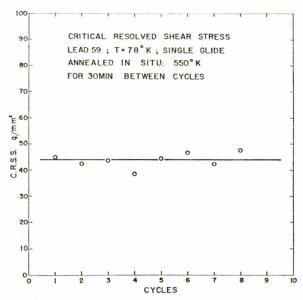


Fig. 2. CRSS of lead single crystal, tested a number of times at 78 °K, with intermediate anneals in situ.

is not the case, since in this investigation specimens which were left at room temperature had appreciably higher values of the CRSS than those annealed at high temperatures, i.e., they were not fully annealed.

To show the effect of prior deformation of the specimen on the CRSS, one specimen, after annealing *in situ*, was bent a small amount at room temperature and the CRSS then determined at 78 °K. The same specimen was then reannealed and the process repeated for different amounts of strain. The results are shown in Fig. 3. Bending the 5-cm specimen 0.8 cm out of the vertical, and back, doubles the CRSS. Further deformation increases this effect.

A series of specimens of 59 and 69 lead, vacuum and air grown, were tested to determine the CRSS after annealing in situ. Specimens were tested several times at 78 °K, or more generally first tested at 78 °K, tested at another temperature, and then back at 78 °K. The purpose of this latter procedure was to try to allow for the scatter between specimens by relating the CRSS at any temperature to that at 78 °K for the same specimen. However, it was found that the scatter in CRSS at 78 °K for a given specimen was comparable in general to the scatter among different specimens, and therefore this procedure was not significant.

The results of these measurements are shown in Fig. 4, for 78 °K and 550 °K. The values for the intermediate temperatures are given in Table I. It is evident from these results that there is no significant difference in the average value of the CRSS for either air-grown or vacuum-grown 59 lead crystals, or between 59 and 69 lead. The average of the air-grown specimens is a little higher than the vacuum-grown, the 69 lead between the two. These differences are not considered significant considering the scatter of the measurements. It is not known

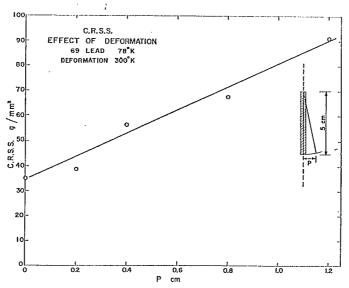


Fig. 3. The effect of prior deformation at 300 °K on the CRSS at 78 °K. One specimen deformed, with intermediate anneals in situ.

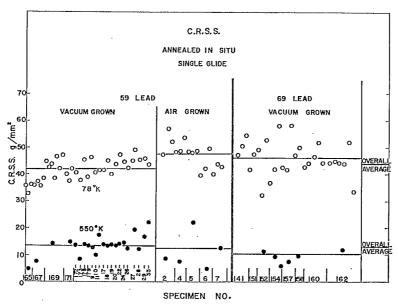


Fig. 4. CRSS of single-glide lead crystals of 59 and 69 purity, grown in vacuum and air, and tested at 78 °K and 550 °K. Specimens annealed in situ, some being tested several times.

TABLE I CRSS (g/mm²), vacuum grown, single glide at various temperatures (°K)

Specimen	78	190	275	325	375	425	475	500	525	550	575
69 Lead Pb64-141 152	See Fig. 4	-	33.6	32.5	25.9	22.2	19,9	14.5	13.8	See Fig. 4	10.8
154 157		34.3	36.0	25.2				12.3	13.3 9.9		
158		$\begin{array}{c} 34.7 \\ 36.7 \end{array}$	24.8	23.2							
160			36.2 27.9 31.2 40.7 33.7 32.3								
162		33,3 41,2 37,0 38,6 39,2	33.8								
59 lead Pb65-24 26						$21.9 \\ 22.6$			14.4		
$\begin{array}{c} 29 \\ 33 \end{array}$			42.0	43.4	46.5	28.0			$\begin{array}{c} 14.4 \\ 18.6 \end{array}$		
Average	43.6	36.9	33.8	31.1	36.2	23,7	19.9	13.4	14.0	13.0	10.8
No. of readings	63	8	11	4	2	4	1	2	5	27	1.

why the 69 lead samples show more scatter than the 59 specimens, or why the average is higher. The high-temperatures values of the CRSS at 550 °K are similar to the 78 °K results in that they show no significant difference for the three cases considered.

The value for the CRSS of lead at 4.2 °K was determined by S. Saimoto at the National Research Council using the tensile testing apparatus of Z. S. Basinski. For these measurements smaller specimens were used having a square cross section of side 0.32 cm and 5-cm gauge length. They were gripped between serrated plates, in the usual fashion, since they were not tapered at their ends. Since these specimens could not be annealed *in situ*, tests on similar specimens were done at 78 °K and 275 °K on the same apparatus as well as the Instron. The results are shown in Fig. 5.

The values of the CRSS for these smaller specimens, annealed *in situ* and tested on the Instron, are shown on the left. It is seen that they are higher than the average value for standard specimens shown on the right. Special grips were not made for these specimens so that the high value is attributed to inadequate alignment. The average value at 78 °K on Basinski's tensile machine agrees very well with the average value of the standard specimens, indicating that there is no size or machine effect and that the values obtained at 4.2 °K can be compared to the standard specimen results. The three results at 275 °K show a

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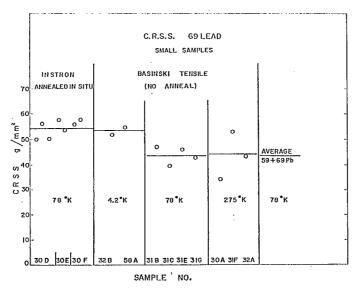


Fig. 5. CRSS of single-glide lead crystals of 0.32×0.32 cm section, tested on Instron and Basinski tensile machines at the temperatures shown.

high average value compared to the standard specimens and more scatter. This is due to the high value obtained for specimen 31F; without this point the results would compare reasonably with the standard specimens.

The effect of growth conditions and gaseous impurities on the CRSS were investigated by (1) cleaning the graphite boat with nitric acid and baking it out, (2) heating the boat at a high temperature in a chlorine atmosphere to remove impurities, and (3) attempting to remove dissolved oxygen from the lead by bubbling hydrogen through the molten lead as well as heating the molten lead to a relatively high temperature in vacuum. In addition the effect of growth striations on the CRSS was also examined by passing a grain boundary through the crystal from one pinched end, using a suitable annealing treatment.

The results are shown in Fig. 6. The CRSS values for the boat cleaned in nitric acid are the same as the average value (shown on the right) indicating no effect resulting from nitric acid cleaning. Passing a boundary through the crystal to eliminate striations significantly lowers the CRSS (note that only one specimen was tested). Using a chlorinated boat also significantly lowers the CRSS to about the same value as the striation-free crystal. Bubbling hydrogen through the molten lead for 5 hours at 420 °C had no observable effect, nor did heating the molten lead *in situ* for 20 hours at 750 °C before growing the crystal. In the latter case roughly half the lead was evaporated from the bath and deposited on the cold ends of the vycor tube containing the boat. The average value of the CRSS under optimum conditions, from Fig. 6, is 34 g/mm² at 78 °K.

The temperature dependence of the CRSS was investigated for single-glide, double-glide, and [100] oriented crystals, as well as for alloy crystals. The results for the single-glide oriented specimens, annealed *in situ* (taken from Table I), are given in Fig. 7, along with the averaged results for the unannealed specimens

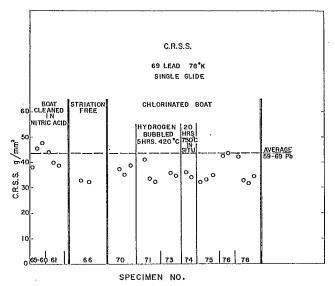


Fig. 6. CRSS of single-glide lead crystals at 78 °K for the conditions shown.

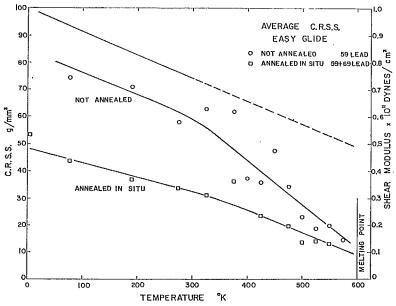


Fig. 7. The top curve shows the temperature dependence of the shear modulus, the middle curve the average value of CRSS of "as-grown" lead crystals (Fig. 1), and the lower curve shows average values of the CRSS of lead crystals annealed in situ.

taken from Fig. 1, and the shear modulus curve. It is immediately apparent that the curve for annealed $in\ situ$ specimens lies well below that for the unannealed specimens and shows much less scatter. The number of values averaged to determine each point varied appreciably, as shown in Table I and Fig. 4. One point at 475 °K is appreciably above the curve. Since only one measurement was made at this temperature, this deviation is not considered significant. The CRSS for the annealed specimens shows a small drop on going from 4.2 to 78 °K, a linear decrease with increasing temperature to roughly 300 °K (0.5 $T_{\rm m}$), followed by a more rapid decrease to the melting point.

To determine the behavior of the CRSS near the melting temperature, four tests were conducted in which the heating-bath temperature was slowly increased as the specimens were strained. Two of the specimens were deformed at a slow rate of $1.7 \times 10^{-5} \, \mathrm{sec^{-1}}$ and two at $1.7 \times 10^{-3} \, \mathrm{sec^{-1}}$. In all cases there was a sharp drop in stress when the specimen melted, indicating that the CRSS does not progressively approach zero near the melting temperature. The two specimens pulled at the lower rate were strained 2.5% when melting occurred. The stress on melting was $13.4 \, \mathrm{g/mm^2}$, which is a little higher than the average CRSS at $550 \, \mathrm{and} \, 575 \, \mathrm{^oK}$ but within the scatter. At the higher strain rates, where specimens were deformed 25%, work hardening was evident and the stress at melting was appreciably higher than that at $585 \, \mathrm{^oK} \, (13.7 \, \mathrm{g/mm^2} \, to 25.5 \, \mathrm{g/mm^2})$. The above results suggest that there is a small drop in the CRSS, commensurate with the general temperature-dependence curve, up to the melting point.

The temperature dependence of the CRSS corrected for the shear modulus temperature dependence, CRSS/ μ , is shown in Fig. 8. It is seen that the modulus-corrected CRSS is independent of temperature in the temperature region of

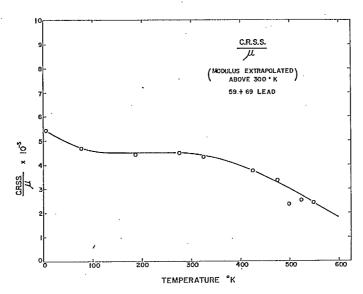


Fig. 8. Temperature dependence of the CRSS/ μ .

100 to 300 °K, increases a small amount below 100 °K, and decreases progressively above 300 °K. This is generally similar to the temperature dependence of the flow stress for copper and aluminium.

The temperature dependence of the flow stress of lead specimens has been determined in another part of the present investigation, and is shown for single-glide and $\langle 100 \rangle$ -oriented specimens in Fig. 9. In the figure, the ratio of loads is plotted, which equals the flow-stress ratio $\sigma_T/\sigma_{78}^{\circ}_{\text{K}}$, as a function of temperature. For comparison, CRSS_T/CRSS₇₈°_K is also plotted.

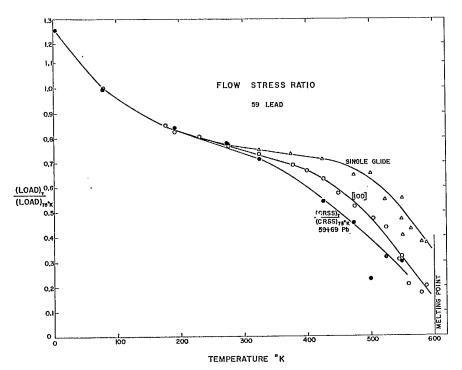


FIG. 9. The top curve shows the flow stress ratio of single glide lead crystals as a function of temperature; the middle curve shows the flow stress ratio for (100) oriented crystals and the lower curve the CRSS ratio temperature dependence.

If the results for aluminium reported by Lucke and Buhler (1963) were applicable to lead, then the flow-stress-ratio curve would coincide with the CRSS-ratio curve at the higher temperatures. From Fig. 9 it is apparent that this is not the case. Below approximately 300 °K the three curves do coincide.

The orientation dependence of the CRSS of lead is shown in Fig. 10. Higher values of the CRSS are obtained at the edges and corners of the stereographic triangle, in general agreement with the results on copper obtained by Diehl (1956).

Specimens oriented near the sides of the stereographic triangle, i.e. oriented for double glide, were tested as a function of temperature. The results are shown in Fig. 11, with the single-glide curve plotted for comparison. The CRSS for

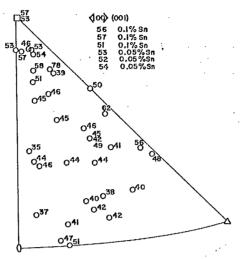
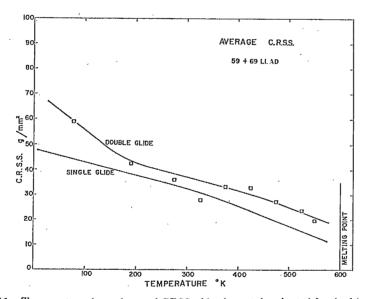


Fig. 10. Orientation dependence of CRSS of 59 \pm 69 lead single crystals at 78 °K. Values of CRSS of $\langle100\rangle$ crystals containing Sn listed.



 F_{1G} . 11. Temperature dependence of CRSS of lead crystals oriented for double glide (upper curve) and single glide (lower curve).

double glide is a little higher than that for single glide, with the same temperature dependence, above roughly 150 °K. Below this temperature region the CRSS increases more rapidly than for the single-glide specimens.

Specimens oriented with a $\langle 100 \rangle$ axis parallel to the tension axis had an average CRSS which varied with temperature in the manner shown by the circles plotted in Fig. 12. The curve for easy glide is plotted for comparison, as well as values for crystals of the same orientation containing 0.05% and 0.1%

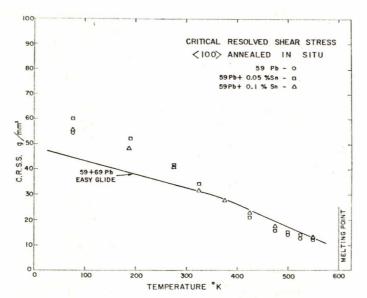


Fig. 12. Temperature dependence of CRSS for $\langle 100 \rangle$ -oriented crystals plotted for 59 Pb and Pb + 0.05 and 0.1% Sn. The solid curve shows the CRSS single-glide 59 + 69 lead crystals.

Sn. Comparing the $\langle 100 \rangle$ -oriented specimens with single glide, it is seen that, as before, the difference becomes more marked at lower temperatures. In this case the $\langle 100 \rangle$ -oriented crystals coincide with the single-glide specimens above roughly 300 °K, and have progressively higher values of CRSS below 300 °K. Considering the alloy crystals, the results indicate that there is no effect on the CRSS with the addition of up to 0.1% Sn.

Alloy crystals oriented for single glide, containing 1.0 wt.% Sn and 0.006% and 0.021% Cu were tested as a function of temperature and the results are shown in Fig. 13. Considering the 1.0% Sn alloy, a very marked increase is observed in the CRSS below approximately 300 °K, which increases with decreasing temperature. Above 300 °K the curve is approximately parallel to the pure lead curve and a little above it. The Pb–Cu alloys exhibit a small increase in the CRSS in the region of 300 °K, increasing with increasing solute concentration. Both above and below this temperature the CRSS tends to approach the pure lead values.

DISCUSSION

It was found that by annealing the lead specimens in situ, the CRSS could be appreciably reduced and the scatter diminished. Since annealing the crystals before mounting in the Instron did not have the same effect, one can conclude that the specimens were strained during handling and mounting. It is surprising that specimens could be completely recovered after 0.1% strain, by annealing, since this is not generally considered possible for f.c.c. metals. Other f.c.c. metals might behave in a similar manner if suitably annealed in situ.

The average value of the CRSS of lead, obtained under optimum conditions, is 34 g/mm² at 78 °K. Since the CRSS is not significantly sensitive to the level of the trace impurities present, to small solute additions, or to the presence of oxygen in the lead, it appears unlikely that the CRSS can be reduced appreciably with further purification of the lead or by improved testing procedures. Accordingly, either expression (1) for the flow stress is not applicable to the CRSS, as defined in this investigation, or the expression should be modified or redefined in some way to account for the high value of the CRSS for lead.

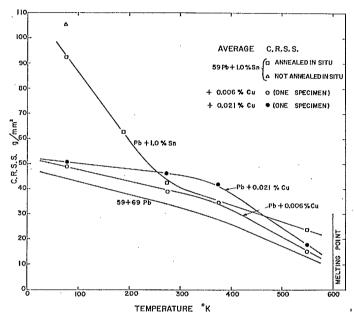


Fig. 13. Temperature dependence of CRSS for single-glide lead crystals containing 1.0% Sn and 0.006 and 0.021% Cu. The solid curve shows the CRSS for easy glide of 59+69 lead crystals.

Considering the first point, Young (1962) has shown in copper that extensive dislocation movement and multiplication occurs during the elastic portion of the stress-strain curve. By the time macroscopic plastic deformation occurs, i.e. at the CRSS, the dislocation density has reached roughly 106 lines/cm² relatively independent of the initial dislocation density of the crystal. Accordingly, assuming that the same process occurs in lead before the CRSS is reached, there is a relatively large dislocation density present in the lead specimens resulting from the applied stress. It is reasonable then to consider the CRSS as a flow stress governed by the same factors as the rest of the stress-strain curve and therefore defined by equation (1).

The high CRSS of lead, as compared to copper, may be attributed to the high stacking-fault energy of lead. Both Seeger (1956) and Nabarro *et al.* (1964) have considered that lead has a high stacking-fault energy, comparable to that of aluminium. The main basis for this conclusion appears to be that lead exhibits

few annealing twins in comparison with copper. However, Bolling *et al.* (1962) have reported a high incidence of annealing twins in pure lead, comparable to that in copper. The present author has also observed extensive numbers of annealing twins in lead. Bolling *et al.* measured the stacking-fault energy of lead, primarily on the basis of the stress at the beginning of stage 3, and reported it to be 24.5 ergs/cm², considerably lower than that of copper.

Nabarro, Basinski, and Holt (1964) point out that the proper parameter to compare when relating work-hardening properties of materials with different stacking-fault energies is $\gamma/b\mu$, where γ is the stacking-fault energy, b is the Burgers vector, and μ is the shear modulus. Values for this parameter are listed in Table II using the values of γ for Cu, Ag, Au, and Ni given by Kronmüller at

TABLE II

	γ (ergs/cm²)	b (Å)	$\mu(\times 10^{11} \text{dynes/cm}^2)$	$\gamma/b\mu~(\times 10^{-3})$
Silver	21±6	2.883	2.56	2,9
Lead	24.5	3.493	0.73	9.6
Gold	43 ± 11	2.878	2.37	2.3
Copper	58±18	2.551	4.08	5.6
Nickel	185 ± 55	2.487	7.46	10
Aluminium	≈200	2.857	2.51	28

this conference, the value for lead determined by Bolling et al. (1962), and an estimate generally used for aluminium. The shear moduli are for room temperature.

It is apparent from Table II that lead should be included with Cu, Ag, Au, and Ni as a material of intermediate stacking-fault energy, following the nomenclature of Nabarro, Basinski, and Holt (1964) and not with Al as a material of high stacking-fault energy.

Assuming that the values for γ used in Table II are reasonably correct, then it is evident that $\gamma/b\mu$ for lead is a little higher than that for copper, the same as that for nickel, and appreciably lower than for aluminium.

The difference between $\gamma/b\mu$ for Au and Cu is roughly equivalent to the difference between Cu and Pb. The CRSS for high-purity Au at 93 °K is 59 g/mm² (Andrade and Henderson 1951), that of Cu approximately 40 g/mm² and Pb 34 g/mm². In the light of the uncertainty in γ and the CRSS, little significance can be attached to the small decrease in the CRSS on going from Au to Pb. If expression (1) for the flow stress is modified to take into account the difference in stacking-fault energy, possibly by making N into an effective dislocation density $N(\gamma/b\mu)$, it is difficult to see how this could be done in order to arrive at essentially the same values of the CRSS for the large differences in moduli.

On the basis of the present observations, it is considered that any attempt to modify equation (1) to include stacking-fault-energy terms or effective dislocation densities would be largely speculative and therefore unwarranted. Further careful measurements of CRSS of different f.c.c. materials, in an attempt to define clearly the significance of the shear modulus and the stacking-fault

energy on the CRSS, should be done. These results might indicate how equation (1) should be modified, or show that the expression is not applicable to the CRSS.

It was pointed out earlier that Lucke and Buhler (1963) found that the temperature dependence of the CRSS for aluminium was very similar to that of the flow-stress ratio of aluminium at higher temperatures. From Fig. 9 it is clear that this is not the case for lead. To determine the activation energy Lucke and Buhler used the strain-rate dependence of the CRSS. This was not done in the present investigation because at the higher strain rates it became increasingly difficult to obtain an unambiguous value of the CRSS using the present extrapolation methods. However, an activation energy for the high-temperature deformation of lead was determined following Conrad and Wiedersich (1960), using flow-stress-ratio and strain-rate-sensitivity data for lead. It was found that the activation energy was higher than that of the self-diffusion of lead by a factor of at least two. Since the slope of the CRSS_T/CRSS_{LN} curve is similar to the flow-stress-ratio curve, it is probable that the activation energy determined from the CRSS data would be too high as well.

From the above it would appear that the temperature dependence of the CRSS is not the same as that of the flow-stress ratio and the activation energy is not the same as that of self-diffusion, for the deformation of lead at higher temperatures.

The orientation dependence of the CRSS of lead, as shown in Fig. 10, is similar to that reported by Diehl (1956) for copper. The magnitude of the increase on going from the center of the stereographic triangle to the outside edges or corners appears to be smaller for lead than copper. A reasonable quantitative comparison is not possible because of the scatter of the copper data.

The CRSS for double glide (Fig. 11) and $\langle 100 \rangle$ multiple glide (Fig. 12) increases with decreasing temperatures at the low-temperature portion of the temperature range considered considerably more than the CRSS for single glide. This effect is similar to that observed with alloy crystals, as for example the Pb + 1.0% Sn alloy of Fig. 13, as well as brass crystals. With either double-glide or $\langle 100 \rangle$ -oriented crystals one would expect a higher density of dislocation barriers on the operative slip system in a given region of the crystal. This would effectively raise the flow stress and therefore the CRSS. At higher temperatures the dislocation barriers would not be created as readily, and dislocations could overcome the obstacles by cross-slip or climb resulting in a small increase in flow stress. At low temperatures cross-slip and dislocation climb would be progressively more difficult with decreasing temperatures, resulting in a progressive increase in the CRSS over and above that due to the shear modulus.

In Fig. 11 it can be observed that above 200 °K the CRSS for double glide is higher than that for single glide, whereas for the $\langle 100 \rangle$ -oriented crystals in Fig. 12 the values coincide above 300 °K. The CRSS curve for double glide is sufficiently close to that for single glide at the higher temperatures that it is considered that little significance should be attached to this difference.

The addition of small amounts of Sn and Cu to Pb has a relatively small effect on the CRSS, as shown in Figs. 12 and 13. For the (100)-oriented crystals there is no observable effect for additions of Sn up to 0.1%. It has been proposed that the effect of solute additions to a material on the CRSS can be estimated from the difference in the Goldschmit atomic diameter of solute and solvent (Honeycombe 1961). The expression given is

$$d\tau_0/dc = K\Delta D^2,$$

where τ_0 is the CRSS, C is the atomic concentration, and K is a constant. The value of ΔD for the Pb-Sn system is 0.33 Å, which turns out to be the same as that of the Cu-Au system. The value for Pb-Cu is 0.97 Å.

An addition of 0.1 wt.% (0.16 at.%) of Sn to Pb should have the same effect as an addition of 0.16 at.% of Au to Cu. From the data reproduced in Honeycombe (1961) τ_0 for 0.16% Cu–Au alloy is 1.7 greater than for pure Cu at room temperature. For the same concentration of Sn in Pb no significant increase in the CRSS is observed because of the addition of the Sn, indicating that the present results do not fit the above theory. The discrepancy is even greater for the Pb–Cu alloy since the value of ΔD is correspondingly larger than for the Pb–Sn system.

The solid solubility of Cu in Pb is less than 0.007 wt.% of Cu (Hansen 1958). Accordingly, in the 0.021% Cu alloy one would expect hardening due to the Cu in solution as well as hardening due to the probable presence of fine precipitates resulting from the excess copper. The actual increase due to the presence of copper was small, being a maximum at 300 °K and a minimum at both low and high temperatures. The low-temperature behavior, in particular, is quite surprising. No explanation for this behavior is apparent at present.

CONCLUSIONS

- 1. Under optimum conditions, the average value of the CRSS for lead is $34 \,\mathrm{g/mm^2} \,\mathrm{at} \, 78 \,\mathrm{^oK}$.
- 2. Comparing this value with that of copper (40 g/mm²) and using the general expression for the flow stress, the value for lead is too high by a factor of four.
- 3. The CRSS of lead is relatively insensitive to the level of impurities in the material and to small solute additions. With larger solute additions the CRSS increases at low temperatures.
- 4. The relatively small effect of solute additions is not in agreement with that predicted on the basis of the difference in atomic diameters of solute and solvent.
- 5. The CRSS decreases with increasing temperature from $54~\rm g/mm^2$ at $4.2~\rm ^{\circ}K$ to approximately $10~\rm g/mm^2$ at the melting point. Between $100~\rm and~300~\rm ^{\circ}K$ the temperature dependence of the CRSS is the same as that of the shear modulus.
- 6. With specimens oriented for double or multiple glide, a significant increase in the CRSS above that of single glide is observed at low temperatures.
- 7. A comparison of the temperature dependence of the CRSS with the flowstress ratio at higher temperatures shows that they do not coincide, differing from the results reported for aluminium.

ACKNOWLEDGMENTS

I would like to thank P. Fryzuk for assistance in the measurements, S. Saimoto of the National Research Council for carrying out the tests at 4.2 °K, Z. S. Basinski for the use of his tensile apparatus, and the Consolidated Mining and Smelting Company for the trace analyses of the lead specimens.

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DISCUSSION

G. B. Craig: The data presented by the author show that the lowest critical resolved shear stresses were found in boundary pass crystals. This treatment eliminates striation-type substructure but higher-order substructures are still present. Variations in size of the higher-order substructures can alter the critical resolved shear stress by a factor of three. Is the large value of the critical resolved shear stress due to the presence of substructure?

Author: The dislocation structures in the lead specimens tested were not determined, since there is no suitable dislocation etch available to the best of my knowledge. All the "as-grown" specimens contained lineage structure as described in the literature.

The contribution that substructure would make to the critical resolved shear stress is not clear for several reasons: (1) Young has reported that extensive multiplication and movement of dislocations occurs long before the onset of easy glide. He observed dislocation densities of the order of 10°/cm² at the onset of easy glide relatively independent of the initial dislocation density and therefore possibly of the initial substructure present. (2) The size and distribution of "as-grown" substructure varies to some extent with orientation. No significant orientation dependence was observed other than the expected small increase in CRSS where multiple slip occurred. (3) In general the "as-grown" substructures have been observed to be highly stable when subjected to prolonged annealing. However, it is not clear what effect a series of deformation cycles of 0.1% at 78 °K separated by annealing treatments would have on the "as-grown" substructure. The present results show that the effects of deformation can be completely recovered, indicating no significant change in the substructure pattern, if the substructure significantly affects the CRSS.

H. Kronmüller: As mentioned in the report, Seeger has proposed in the Lake Placid Report a large stacking-fault energy for lead. This is in fact compatible with your result. The question whether γ is large or small is answered by calculating the ratio γ/Gb , which you found to be equal to 9.6×10^{-3} . In comparison with copper $(\gamma/Gb \sim 5 \times 10^{-3})$ the stacking-fault energy of lead seems to be quite large.

Author: In both Seeger's report and in the Nabarro, Basinski, and Holt review article, lead is grouped with aluminium $(\gamma/Gb = 34 \times 10^{-3})$ as a high-energy stacking-fault material and copper, silver, and gold $(\gamma/Gb \approx 5 \times 10^{-3})$ are listed as having an intermediate stacking-

fault energy. Considering the uncertainty in the values of γ quoted for all the materials, it is suggested that lead should be included in the intermediate category and not grouped with aluminium.

- G. F. Bolling: It seem worthwhile noting that values of $\gamma(\text{Pb}) \sim 25$ ergs cm⁻² are consistent with several different types of published observation: twin-boundary energy, annealing twin frequency, (X-ray) stacking-fault probability, " τ_{HI} ", and, most recently, mechanical twinning at 77 °K.
- **D. K. Wilsdorf:** Dr. T. R. Duncan, in his Ph.D. research at the University of Virginia, has calculated the stresses and energies of $a/2(110)\{111\}$ dislocations in cubic anisotropy. His calculations show that the common corrections of the elastic modulus in order to take account of anisotropy, such as Dr. Weinberg has used, are satisfactory as far as energies of dislocations are concerned. However, the maximum shear stresses due to dislocations, which quite likely represent the important parameter in the context of Dr. Weinberg's contribution, are not at all well represented. Now I have just checked our results and found that the discrepancy between the yield stresses of lead and copper would be still enhanced by this effect of anisotropy, by perhaps about 20%.
- J. S. Nadeau: There once appeared to be a good correlation between the critical resolved shear stress of alkali halides and their shear moduli. As the purity of alkali halide crystals improved, however the correlation broke down. Now a log G vs. log CRSS plot shows a random scattering of points; if a line of slope unity is drawn through the lowest point, which is LiF, some of the alkali halides are found to be several times harder than would be expected.

