Submitted to Metallurgical
Transactions

LBL-831
Preprint

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May 1972

AEC Contract No. W-7405-eng-48

LBL-831

DISCLAIMER

HARPER-DORN CREEP IN ALUMINUM, LEAD AND TIN

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ABSTRACT

High-temperature creep has been studied in lead and tin in the stress range of 10^{-6}G to 10^{-4}G using double-shear type specimens in a range of temperatures to near the melting point. A composite plot of dimensionless parameter $\frac{\dot{\gamma}kT}{DGb}$ versus τ/G revealed transition from high stress region (with slope $\sim$4.9 for lead and $\sim$6.6 for tin) to low stress region (with slope $\sim$1 for both metals), at stresses of $\sim 2 \times 10^{-5}$ G for lead and 10^{-5} x G for tin. The creep behavior in the low stress region is identical to that attributable to Harper-Dorn mechanism. In addition, the earlier work on aluminum in the low stress region is confirmed using polycrystalline as well as single crystal samples.

1. INTRODUCTION

In studies on the steady state creep of pure aluminum in the limit of low stress and high temperature Harper and Dorn[1,2] found an anomalous creep behavior which shows two principal characteristics: (1) the steady-state strain rate increases linearly with stress at constant temperature; (2) the steady-state strain rate at given temperature and applied stress is independent of grain size. The second characteristic of the Harper-Dorn data indicated the existence of a new mechanism of steady-state creep. Since the strain rate does not depend explicitly on grain size the classic Nabarro-Herring mechanism of diffusional creep cannot be used to explain the linear relation between strain rate and applied stress.

Recently Murty, Mohamed and Dorn[3] found behavior of the Harper-Dorn type in low-stress, high temperature creep of Al-3% Mg alloy, showing that Harper-Dorn creep is not peculiar to pure aluminum. On the other hand, Muehleisen[4] found no evidence for behavior of the Harper-Dorn type in low-stress creep of pure copper, although he was able to confirm the results of Harper and Dorn on aluminum. These results raised the possibility that Harper-Dorn creep is unique to aluminum alloys.

This possibility motivated the work reported here, in which we investigated the low stress, high temperature creep of pure lead and tin. These two metals were chosen because of their low melting points (allowing good temperature control in tests at temperatures near T_m) and their differing crystal structures (lead is face-centered cubic, tin is body centered tetragonal). Both metals were found to exhibit a creep behavior which appears to be of the Harper-Dorn type.

Since the load and sample geometrics employed in these creep tests differ significantly from those used in the earlier work[1,4] we also measured creep

rates for bulk samples of pure aluminum to test consistency.

2. EXPERIMENTAL TECHNIQUES

Ultra high purity lead and tin (99.999%) was procured from the United Mineral and Chemical Corporation. High purity aluminum was obtained from Cominco American Incorporated. Single crystals of aluminum were grown in this laboratory using a modified Bridgman Technique. The experimental materials were machined into samples of the "double-shear" type described by Murty, Mohamed, and Dorn[3]. Prior to testing the specimens were annealed for the dual purpose of relaxing internal stress and stabilizing the grain size. The lead samples were annealed at 590°K for 48 hours, resulting in a mean grain size of approximately 1.5 mm. The tin samples were annealed for 96 hours at 498°K, yielding a mean grain size of 2mm. The polycrystalline aluminum samples were annealed for 5 days at 927°K, giving a grain size of about 9mm.

The general test procedure employed in these studies is described in ref. 3. Tests on lead and tin were conducted in stirred silican oil baths and the temperatures were maintained to $\pm$ 1°K,,while aluminum tests were conducted in air in an electric furnace. The length changes were recorded by a linear variable differential transformer, and were accurate to $\pm$ 5 x 10^{-5} in. For aluminum the range of stresses from $\sim$1 to $\sim$35 psi was scanned at a temperature of about 923°K ($\sim$0.99 T_m). Three single crystal specimens were used to cover the Harper-Dorn region. In the case of lead, the range of stresses from $\sim$1 to $\sim$100 psi was investigated at two temperatures: 587°K (0.98 T_m) and 554°K (0.93 T_m). Tests at two additional temperatures were conducted to determine the activation energy for creep. During the tests at higher stresses we encountered complications due to sample recrystallization. In this case the minimum strain-rate was taken as the steady-state value as discussed by ref. 5.

This may have caused the relatively high scatter in the lead data. For tin, the stress-range from ∿5 to ∿100 psi was scanned at 495°K (0.98 T_m). Tests at three additional temperatures were conducted for use in determining the activation energy.

3. EXPERIMENTAL RESULTS

Creep Behavior of Aluminum

The results of creep tests on pure aluminum are plotted in Fig. 1 together with the results obtained by Harper and Dorn[1]. The parameters used for this plot are a non-dimensional strain rate, $\dot{\gamma}kT/DGb$, and a non-dimensional stress, (τ/G), where $\dot{\gamma}$ is the steady state shear strain rate, k is Boltzman's constant, T is absolute temperature, D is the self-diffusion coefficient, b is the Burgers vector, G is the shear modulus, and τ is the shear stress. The Harper-Dorn data were rephrased in terms of these variables by transforming the tensile stresses and tensile strains they reported according to the relations

$$\dot{\gamma} = \frac{3}{2}\,\dot{\varepsilon}$$
$$\tau = \frac{1}{2}\,(\sigma - \sigma_o) \qquad [1]$$

The value σ_o appearing in the second of these relations is a correction term Harper and Dorn found necessary to account for an apparent back stress presumedly due to surface effects in their thin platelet samples. Since the present research used bulk samples, no correction term is necessary; the data extrapolate naturally to zero strain rate at zero applied stress.

As is apparent from Fig. 1, the agreement between the present results and those of Harper and Dorn[1] is excellent both in the Harper-Dorn creep region at low stress and in the conventional creep region beyond the knee of the curve. The single and polycrystalline data lie on the same curve,

showing the independence of creep rate and grain size. For comparison, the predicted rate of creep via the Nabarro-Herring mechanism[6,7] has also been plotted, using the relation

$$(\dot{\gamma}kT/DGb) = B\ (b/d)^2\ (\tau/G) \qquad [2]$$

where B is a constant equal to approximately 14. The measured values for low-stress creep in aluminum lie several orders of magnitude above the Nabarro-Herring estimate.

Primary creep was noted in all tests conducted during this research, both in single crystal and polycrystalline samples. This observation also argues with the results of Harper and Dorn.

Creep Behavior of Lead and Tin

(i) Stress Dependence of Creep Rate

The experimental results obtained from isothermal creep tests at 4 different temperatures are presented in Figs. 2(a) and 2(b) for lead and tin respectively. Because of the experimental limitation on the measurement of shear strain-rates below about 2×10^{-9} sec^{-1}, the entire range of stresses could be scanned only at higher temperatures. The data at these higher temperatures do indicate a transition from a higher slope ($\sim$4.9 for lead and $\sim$6.6 for tin) to a lower one ($\sim$1 for both metals). The transition from high stress region to low stress region was observed at shear stress values of $\sim$14.5 and $\sim$16.0 psi for lead at 0.98 T_m and 0.93 T_m respectively. A value of $\sim$32 psi was found for this transition-stress at 0.98 T_m for tin.

(ii) Creep Curves

Typical creep curves observed in both the regions are shown in Figs. 3(a) and 3(b) for lead and tin respectively. As is clear from these, normal primary

creep regions were noted in the entire stress range for both the metals. It is important to note the presence of this primary creep in Harper-Dorn creep region (where the creep-rate is proportional to the applied stress), since the diffusional creep mechanisms[6,7,8] which predict linear creep behavior are characterized by the absence of this primary creep region.

(iii) Temperature Dependence of Creep Rate

To determine the activation energy for creep, the logarithm of the modulus-compensated steady-state strain-rate ($\dot{\gamma}G^{n-1}T$) versus the reciprocal of the absolute temperature was plotted at constant stresses of ∿30 psi and ∿100 psi for lead and tin respectively (Fig. 4). Here values of ∿4 and ∿6 are used respectively for lead and tin for (n-1), since the averages of the slopes of the lines in Figs. 2(a) and 2(b) were found respectively to be ∿5 and ∿7. These data for both the metals fall in the high stress region. The least square analyses of the data presented in Fig. 4 yielded values, for the activation energy for deformation, of 22,920 $\pm$ 180 Cal/mole for lead and 23,070 $\pm$ 1,820 Cal/mole for tin. These are in essential agreement with the self diffusion data, namely

$$D = 0.28 \exp(-24{,}200/RT) \qquad \text{for lead}^{9}$$

and

$$D = 0.78 \exp(-22{,}350/RT) \qquad \text{for tin}^{10}$$

No attempt was made to determine the activation energy for creep in the low stress region. However, a value of 24,945 Cal/mole was inferred from the data at temperatures 587°K and 554°K presented in Fig. 2(a). Thus the activation energy for creep appears to be essentially identical to that for volume self-diffusion throughout the employed stress range. Similar findings were

reported earlier in other metals[1,3].

The creep data for lead and tin presented in Figs. 2 are analysed in terms of the equation,

$$\frac{\dot{\gamma}kT}{DGb} = A\ (\tau/G)^n \qquad [3]$$

and Fig. 5 is a plot of log $\frac{\dot{\gamma}kT}{DGb}$ versus log (τ/G) for both the metals. As is clear from this figure, the data at various temperatures coalesced into a single line for each metal. Again the data reveal transitions from high stress region to low stress region at stresses of $\sim 10^{-5}G$ for tin and $2 \times 10^{-5}G$ for lead. Values of A and n obtained from the least squares analyses of the data are tabulated in Table I, along with the values predicted from the governing equation of Nabarro-Herring creep (Eq. [2]). Again the strain rates lie well above the Nabarro-Herring prediction.

4. DISCUSSION

The experimental results on the steady-state creep behavior of aluminum (poly and single crystals), lead and tin reveal the presence of two parallel creep mechanisms (Fig. 4). The data divide into two regions

(i) High-Stress Region

This region illustrates creep behavior generally attributed to climb-controlled process. Bird, Mukherjee and Dorn[11] made a systematic evaluation of the available creep data and concluded that many aspects of high temperature creep can be correlated with the dimensionless Eq. [3]. According to their analysis, A might be a universal constant with a value of about 10^8 while n, the stress exponent, might be function of stacking fault energy. The present data on aluminum in this stress range are in line with their predictions as well as the earlier work by Servi et al[12], Weertman[13] and Sherby et al[14]. For

lead, their correlation suggested an exponent n of the order of 4.7, compared to the present experimental value of 4.9.

Earlier studies by Dorn and co-workers[15] on the creep of tin indicate that the activation energy for creep is about 22,700 Cal/mole, while Breen and Weertman[16] found a value of 26,000 Cal/mole for the same. These values are in essential agreement with the present one of 23,070 Cal/mole as well as the more recent value for self diffusion in polycrystalline tin[10]. A re-evaluation of the data of Breen and Weertman at higher temperatures where a value of 26,000 Cal/mole was obtained for the activation energy, yielded a value of $5.91 + 0.31$ for n and $(1.03 \begin{smallmatrix} + 14.74 \\ - 0.96 \end{smallmatrix}) \times 10^{15}$ for A. These values should be compared with 6.59 and 3.55×10^{17} obtained in the present investigation. The fact that the above values of A for tin are orders of magnitude larger than those based on the predictions by Bird et al on fcc, Bcc, and HCP metals, illustrates that yet unknown factors concerning the crystal structure also play a significant role in high temperature-creep.

(ii) Low Stress Region

In this region ($\tau/G \leq 10^{-5}$) the stress exponents were found to be near unity for all the three metals (viscous behavior). Mechanisms which predict viscous deformation such as Nabarro-Herring[6,7] and Coble[8] yield values for the strain-rates which are orders of magnitude lower than the observed values. However, as shown in Fig. 6, the present results fall very close to the earlier observations on Al[1] and Al-3% Mg[3]. The present experimental work on bulk samples of aluminum in shear confirm the earlier work by Harper and Dorn who tested thin platelets of both single and polycrystalline aluminum samples in tension. In spite of the difference in the kind of specimen used, the type of stressing, and the grain size of polycrystalline specimens ($d \sim 9$mm in our

investigation, and 3mm in Harper and Dorn study), the agreement between these distinct investigations is excellent.

Although the mechanical data presented here on various metals are consistant with the belief that a dislocation mechanism is responsible for Harper-Dorn creep, it is nevertheless admitted that the dislocation mechanism is uncertain.[1,3,4] However, a good start has been made in determing the special substructural features attending creep by Harper-Dorn mechanism. X-ray topographic, etch pit, and electron transmission microscopy studied in deformed aluminum[4] in this low stress region revealed distinct subgrain formation which presumedly leads to the observed normal primary creep regions in all these metals. Despite the observation that the subgrains do form during the creep in the limit of low stresses, there are no firmly established systematic data regarding the stress dependence of the subgrain size. Moreover, the results indicate that the dislocation density (ρ) is low of the order 10^3 - 10^4 cm^{-2}, and independent of the applied stress. Additional evidence for the stress-independence of dislocation density in aluminum for the same range is obtained from a recent x-ray topographic study in situ during deformation by Nost and Nes[17]. All these results are recorded in Fig. 7 as dimensionless parameter $\sqrt{\rho}\ b$ versus the normalized stress τ/G. At high stresses ($\tau/G \geq 10^{-5}$) the dislocation density increases as $\tau^{2.5}$. The transition in the stress dependence of the dislocation density is extrapolated to be at a stress value of about 3×10^{-6} G. This finding is in agreement with the observed stress level ($\sim 4 \times 10^{-6}$G) in aluminum, where the transition from the climb controlled to Harper-Dorn creep occurs. More needs to be learned about the substructural details, such as the type of dislocations responsible for such creep behavior, and subgrain size. Research toward this goal is currently underway.

CONCLUSIONS

Our principal conclusion from this work is that Harper-Dorn creep is not confined to aluminum and its alloys. A creep behavior which appears to be of the Harper-Dorn type is observed in pure lead and in pure tin over a range of normalized stress (τ/G) roughly identical to the range over which this type of creep is found in aluminum. In addition, our experimental results on low stress, high temperature creep of aluminum reproduce the data of Harper and Dorn almost exactly, despite a significant difference in the sample and load geometries used.

ACKNOWLEDGEMENTS

This work reported here was supported by the United States Atomic Energy Commission through the Inorganic Materials Research Division of the Lawrence Berkeley Laboratory.

REFERENCES

1. J. G. Harper and J. E. Dorn, Acta. Met., 1957, Vol. 5, p. 654.
2. J. G. Harper, L. A. Shepard and J. E. Dorn, Acta. Met., 1958, Vol. 6, p. 507
3. K. L. Murty, F. A. Mohamed and J. E. Dorn, LBL Report #442, December 1971, Acta. Met., (in press).
4. E. C. Muehleisen, Ph.D. Thesis, Stanford University 1969.
5. A. K. Mukherjee, J. E. Bird and J. E. Dorn, "Experimental Correlations for High-Temperature Creep", presented at the Detroit Materials Engineering Congress of ASM, 1968, p. 16.
6. F. R. N. Nabarro, Report of a conference on the strength of solids, The Physical Society, London, 1948, p. 75.
7. C. Herring, J. Appl. Phy., 1950, Vol. 21, p. 437.
8. R. L. Coble, J. Appl. Phys., 1964, Vol. 34, p. 1679.
9. N. Nachtrieb and G. Handler, J. Chem. Phys., 1955, Vol. 23, p. 1569.
10. W. Lange, A. Hassnet and I. Berthold, Phys. Status Solidi (Germany), 1961, Vol. 1, No. 1, p. 50.
11. J. E. Bird, A. K. Mukherjee and J. E. Dorn, "Correlations Between High-Temperature Creep Behavior and Structure," in Quantitative Relation Between Properties and Microstructure, Israel University Press, Jerusalem, 1969.
12. I. S. Servi and N. J. Grant, Trans. AIME, 1951, Vol. 191, p. 909.
13. J. Weertman, J. Mech. and Phy. Solids, 1956, Vol. 4, p. 230.
14. O. D. Sherby, J. L. Lytton and J. E. Dorn, Acta. Met., 1957, Vol. 5, p. 219.
15. R. F. Frenkel, O. D. Sherby and J. E. Dorn, Acta. Met., 1955, Vol. 3, p. 470.
16. J. E. Breen and J. Weertman, Trans. AIME, 1955, Vol. 207, p. 1230.
17. B. Nost and E. Nes, Acta Met., 1969, Vol. 17, p. 13.

FIGURE CAPTIONS

Fig. 1 Plot of log $\frac{\dot{\gamma}kT}{DGb}$ vs. log τ/G for pure aluminum. Data of Harper and Dorn[1] are included for comparison.

Fig. 2(a) The effect of stress on the steady-state creep rate of lead.

Fig. 2(b) The effect of stress on the steady-state creep rate of tin.

Fig. 3(a) Typical creep curves for lead observed in high-stress region (τ = 46.4 psi, T = 554° K) and low-stress region (τ = 7.3 psi, T = 554° K).

Fig. 3(b) Typical creep curves for tin observed in high-stress region (τ = 126 psi, T = 461° K) and low-stress region (τ = 10.8 psi, T = 495° K).

Fig. 4 Effect of temperature on the steady-state creep rate of lead and tin in high stress region.

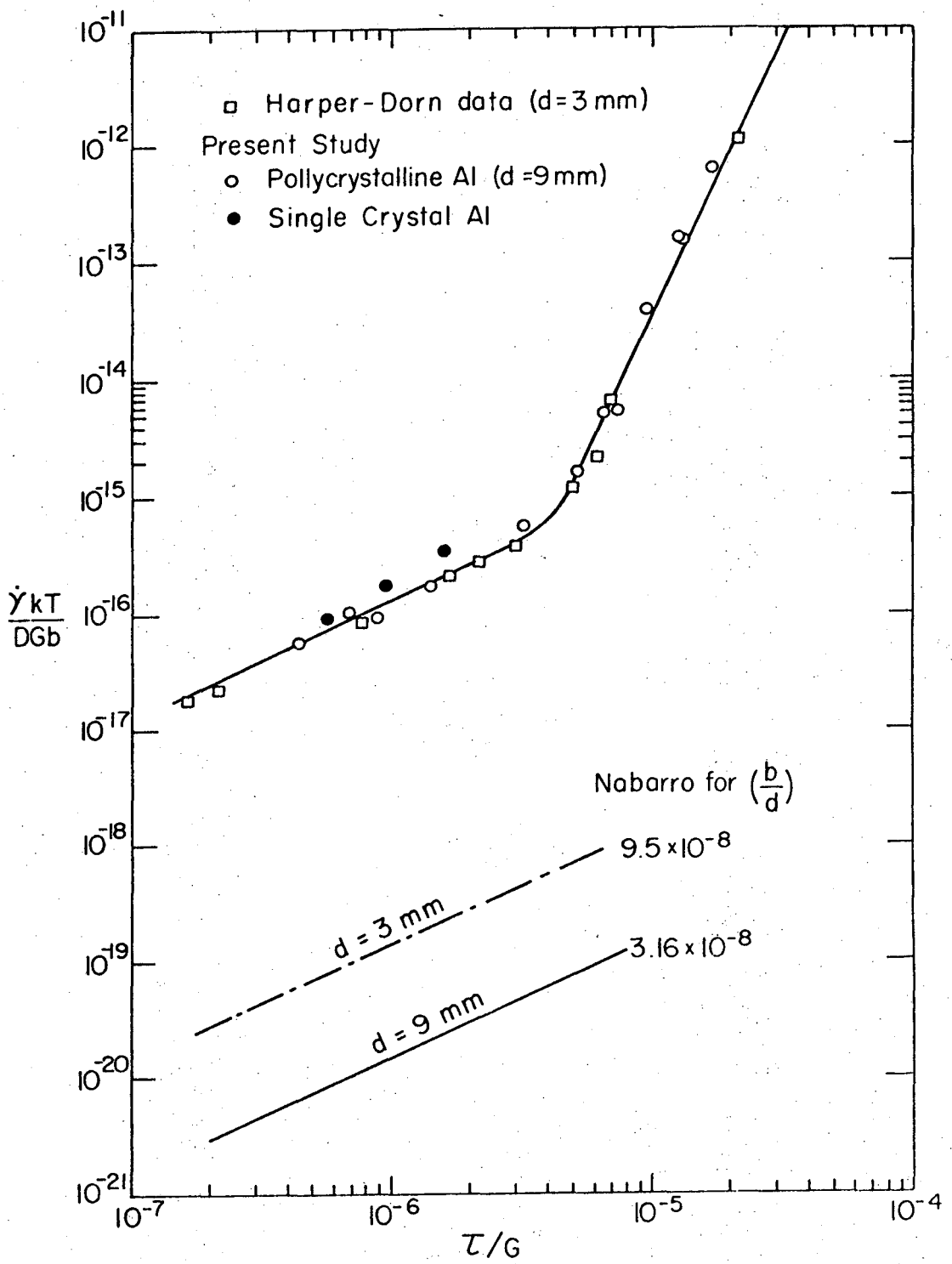
Fig. 5 Plot of log $\frac{\dot{\gamma}kT}{DGb}$ vs. log τ/G for lead and tin, indicating the transition from climb to Harper-Dorn mechanism.

Fig. 6 Data on creep of lead and tin compared to creep behavior of aluminum and aluminum - 3% Magnesium[3].

Fig. 7 Stress dependence of dislocation density.

TABLE I

Metal	Region I		Region II		Nabarro	
	n	A	n	A	n	A
Lead (d = 1.5mm)	0.97 ± 0.88	$(2.88 \, {}^{+\,4.88}_{-\,1.81}) \times 10^{-11}$	4.92 ± 0.57	$(1.38 \, {}^{+\,405,62}_{-\,1.375}) \times 10^{8}$	1	8×10^{-13}
Tin (d = 2mm)	0.94 ± 0.77	$(7.24 \, {}^{+\,9.76}_{-\,4.22}) \times 10^{-11}$	6.59 ± 0.28	$(3.55 \, {}^{+\,59.55}_{-\,3.35}) \times 10^{17}$	1	3.15×10^{-13}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Present Study
Pollycrystalline Al (d =9 mm)
Single Crystal Al
Nabarro for (b/d)
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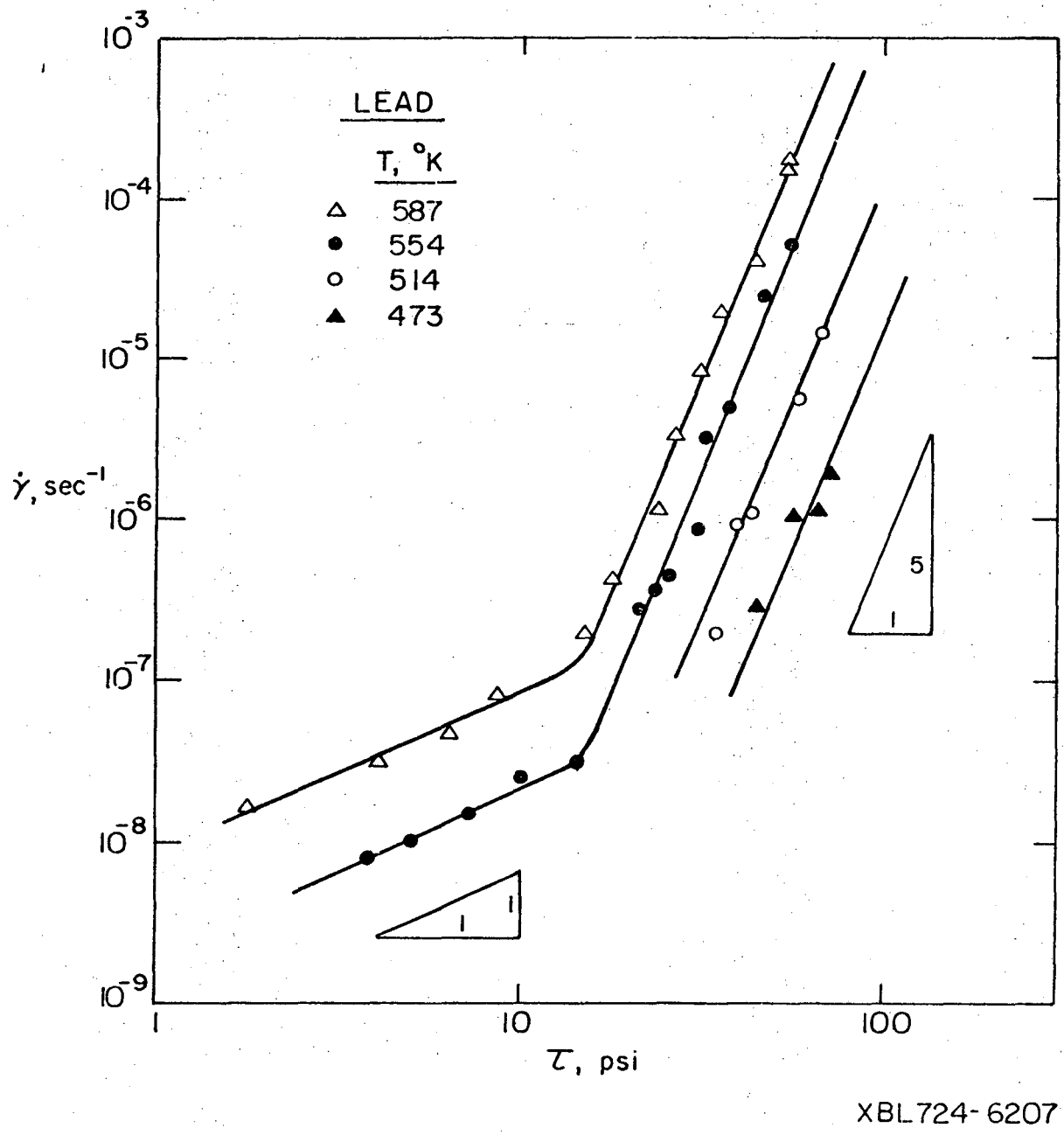
d = 3 mm
3.16 x 10^-8
d = 9 mm
10^-11
10^-12
10^-13
10^-14
10^-15
10^-16
10^-17
10^-18
10^-19
10^-20
10^-21
10^-7
10^-6
10^-5
10^-4
τ/G
Ẏ kT / DGb
XBL724-6178A

Fig. 1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554
514
473
10^{-3}
10^{-4}
10^{-5}
10^{-6}
10^{-7}
10^{-8}
10^{-9}
$\dot{\gamma}$, sec^{-1}
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10
100
τ, psi
5
1
1
1
XBL724-6207

Fig. 2 a

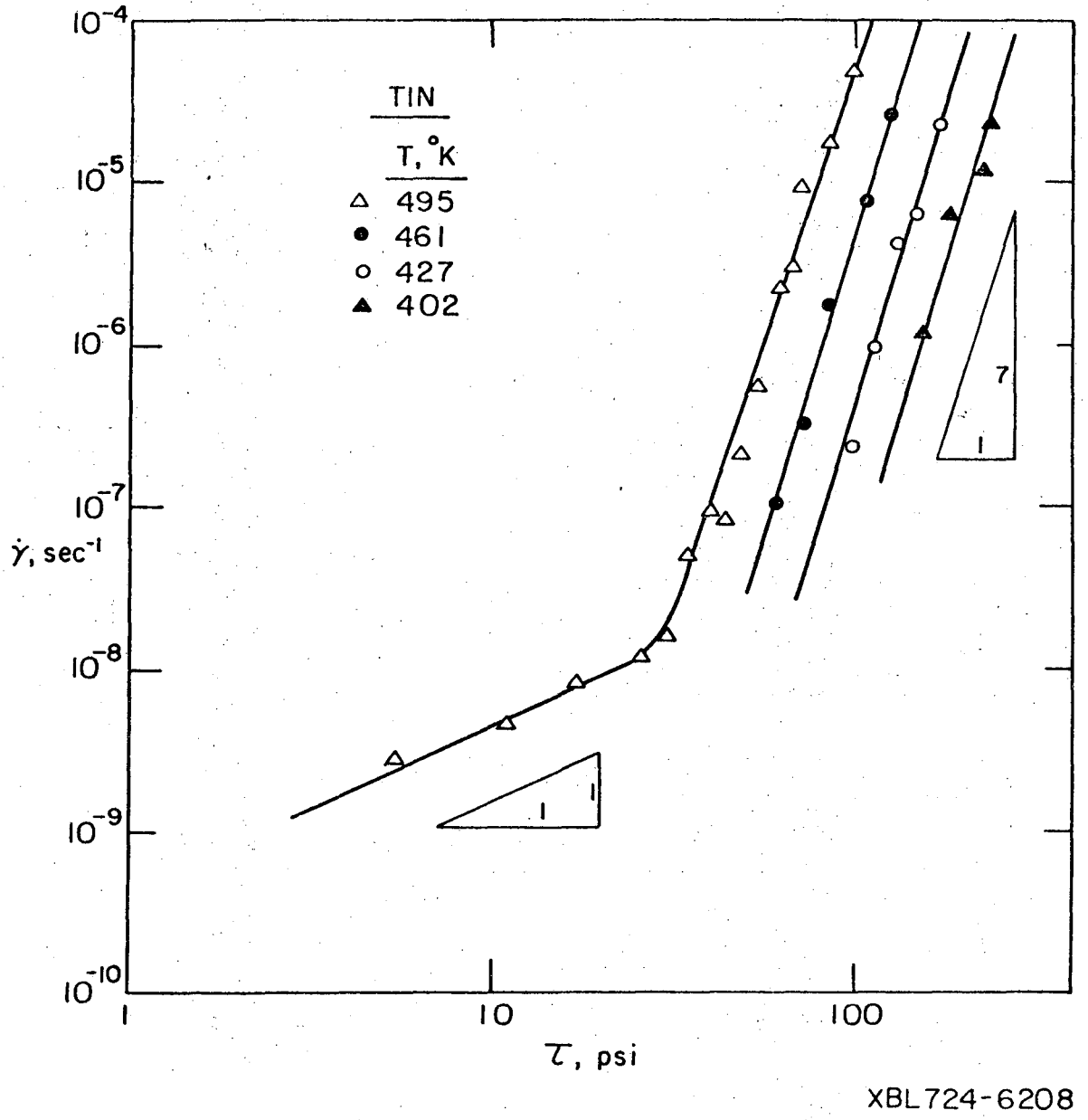
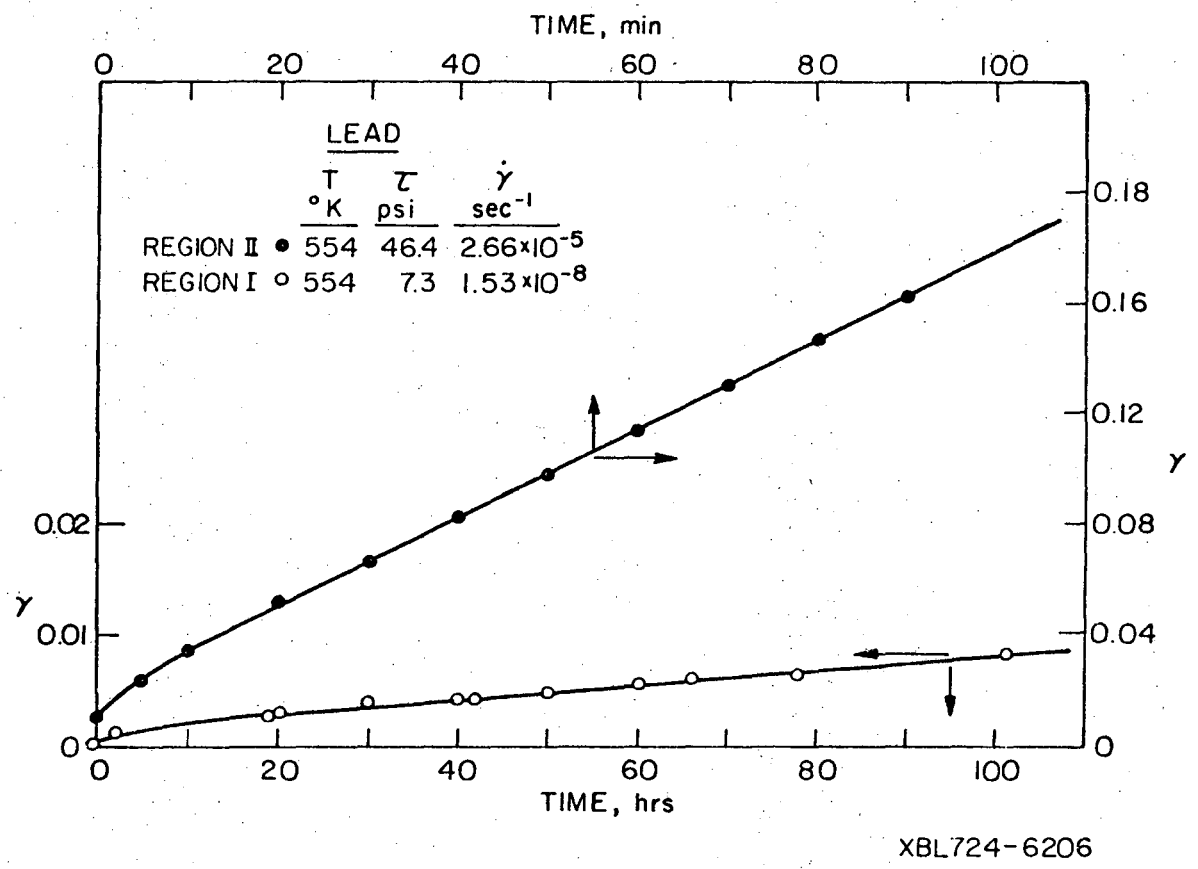


Fig. 2b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40
60
80
100
LEAD
T °K
τ psi
γ̇ sec⁻¹
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REGION I ○ 554 7.3 1.53×10⁻⁸
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γ
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TIME, hrs
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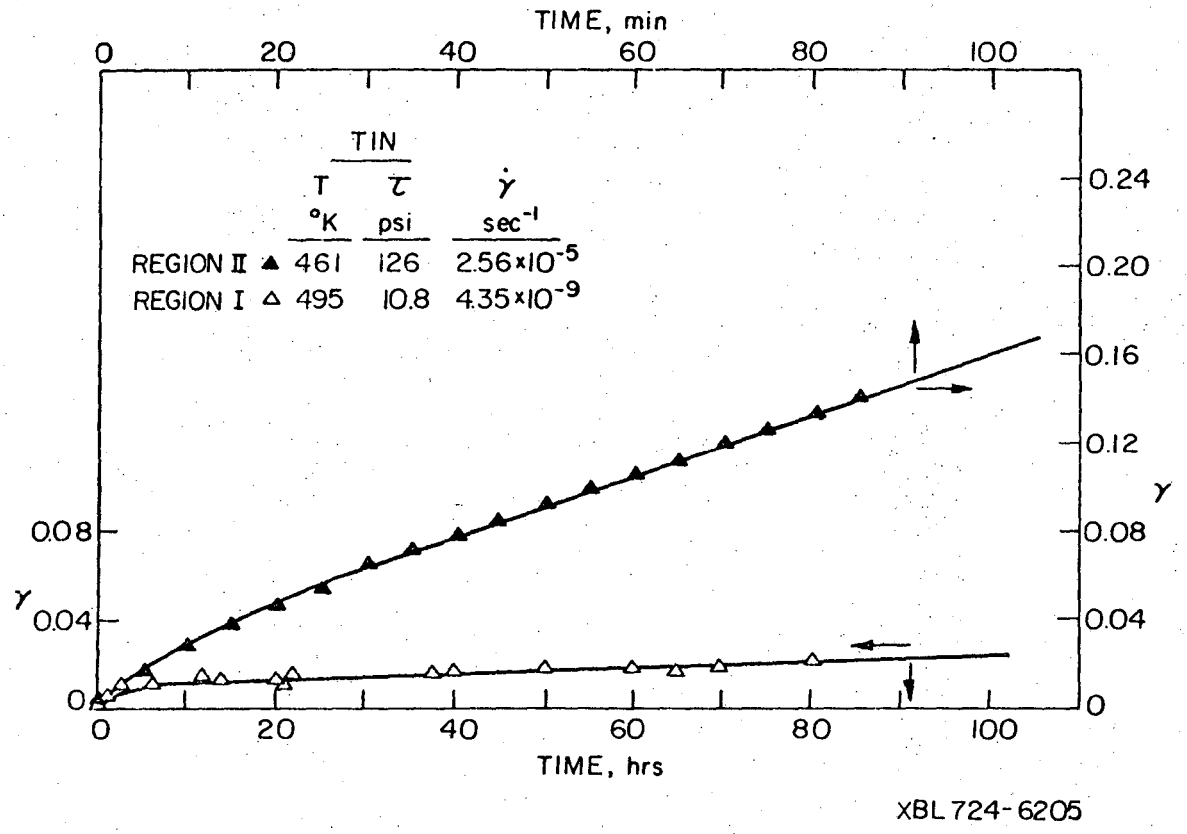
Fig. 3a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TIN
T
°K
τ
psi
γ̇
sec⁻¹
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REGION I △ 495 10.8 4.35×10⁻⁹
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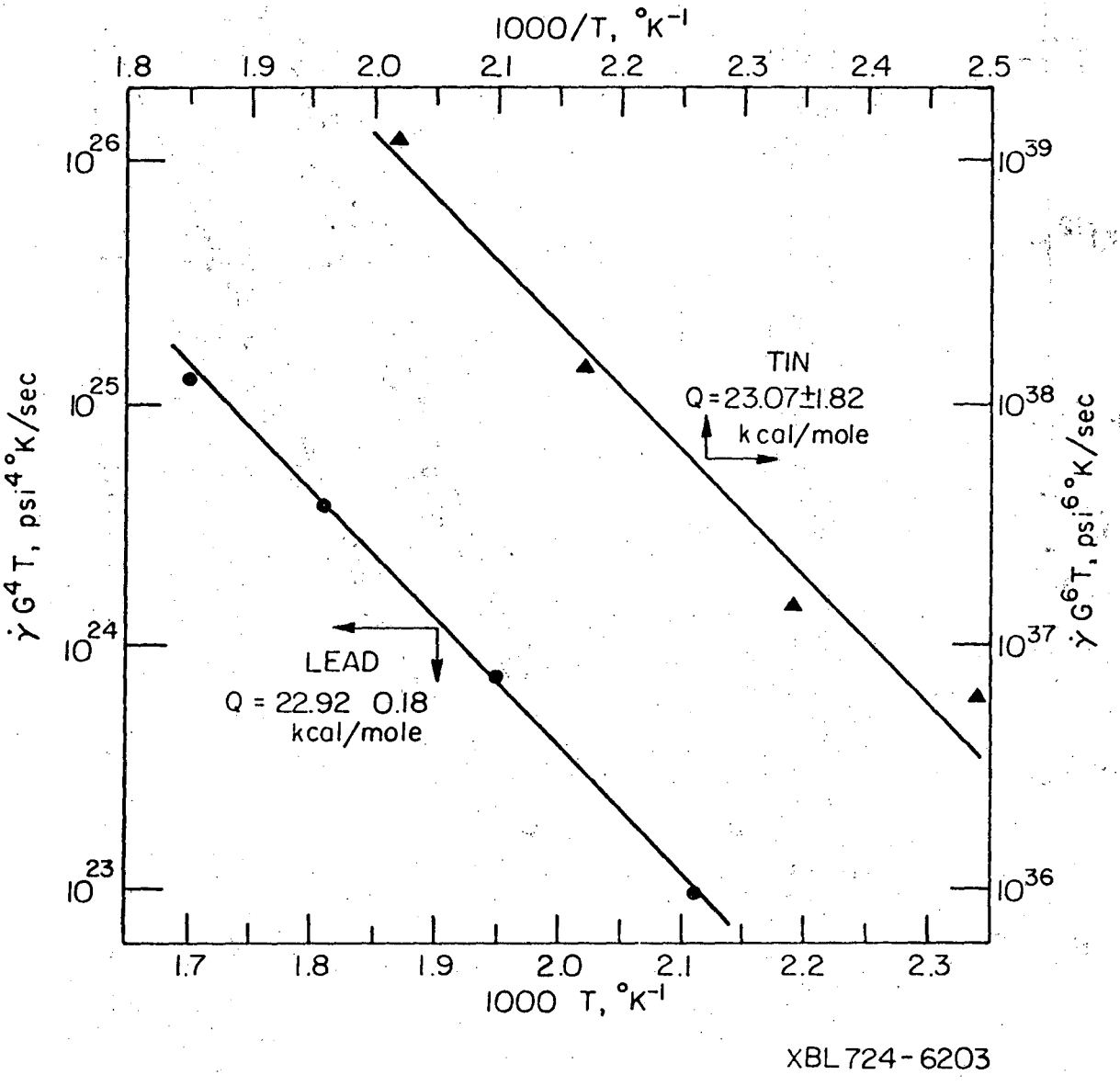
0.12
0.08
0.04
0
γ
TIME, hrs
XBL 724-6205

Fig. 3b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1.9
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2.1
2.2
2.3
2.4
2.5
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10²⁴
10²³
10³⁹
10³⁸
10³⁷
10³⁶
TIN
Q=23.07±1.82
k cal/mole
LEAD
Q = 22.92 0.18
kcal/mole
γ̇ G⁴ T, psi⁴ °K/sec
γ̇ G⁶ T, psi⁶ °K/sec
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1000 T, °K⁻¹
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Fig. 4

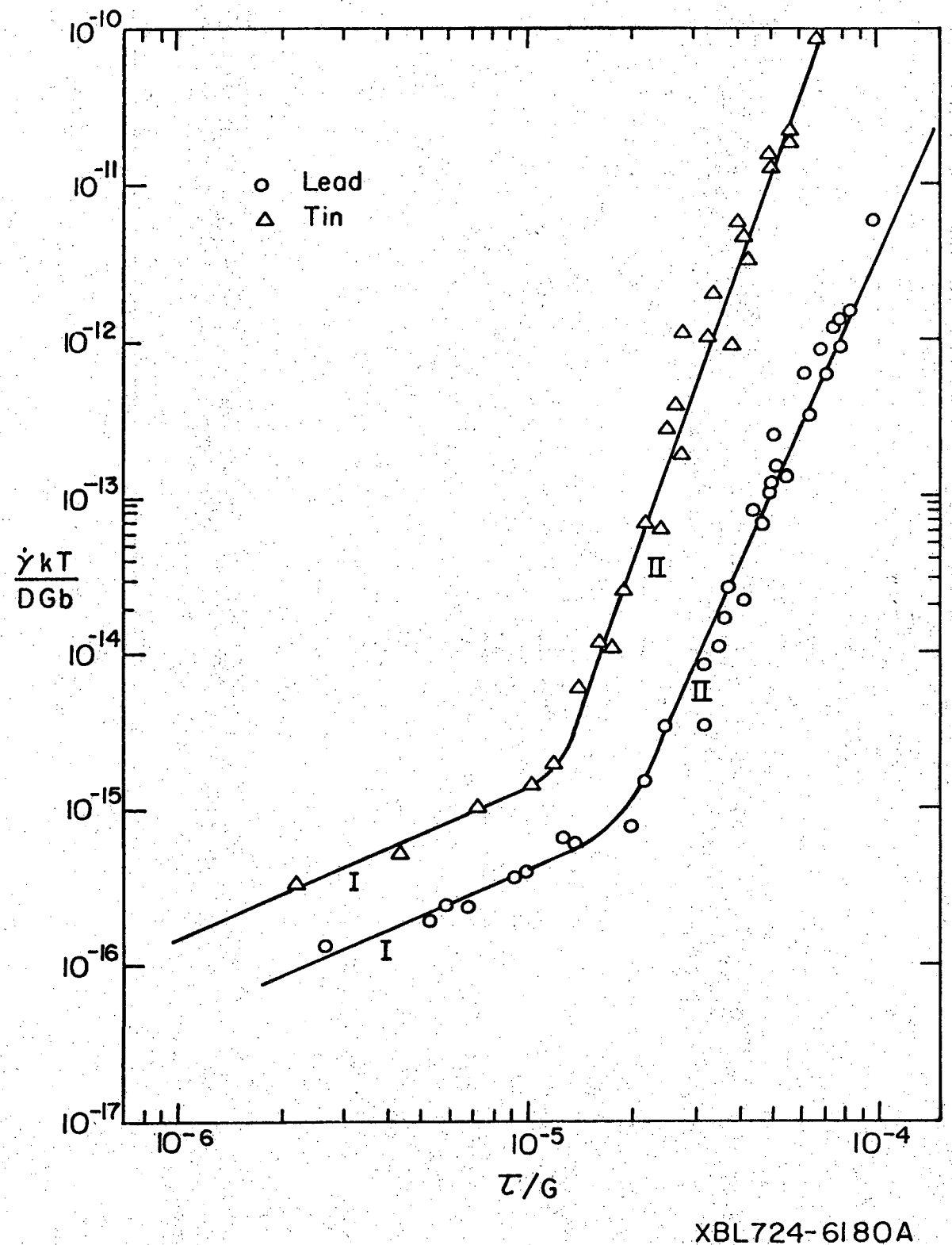
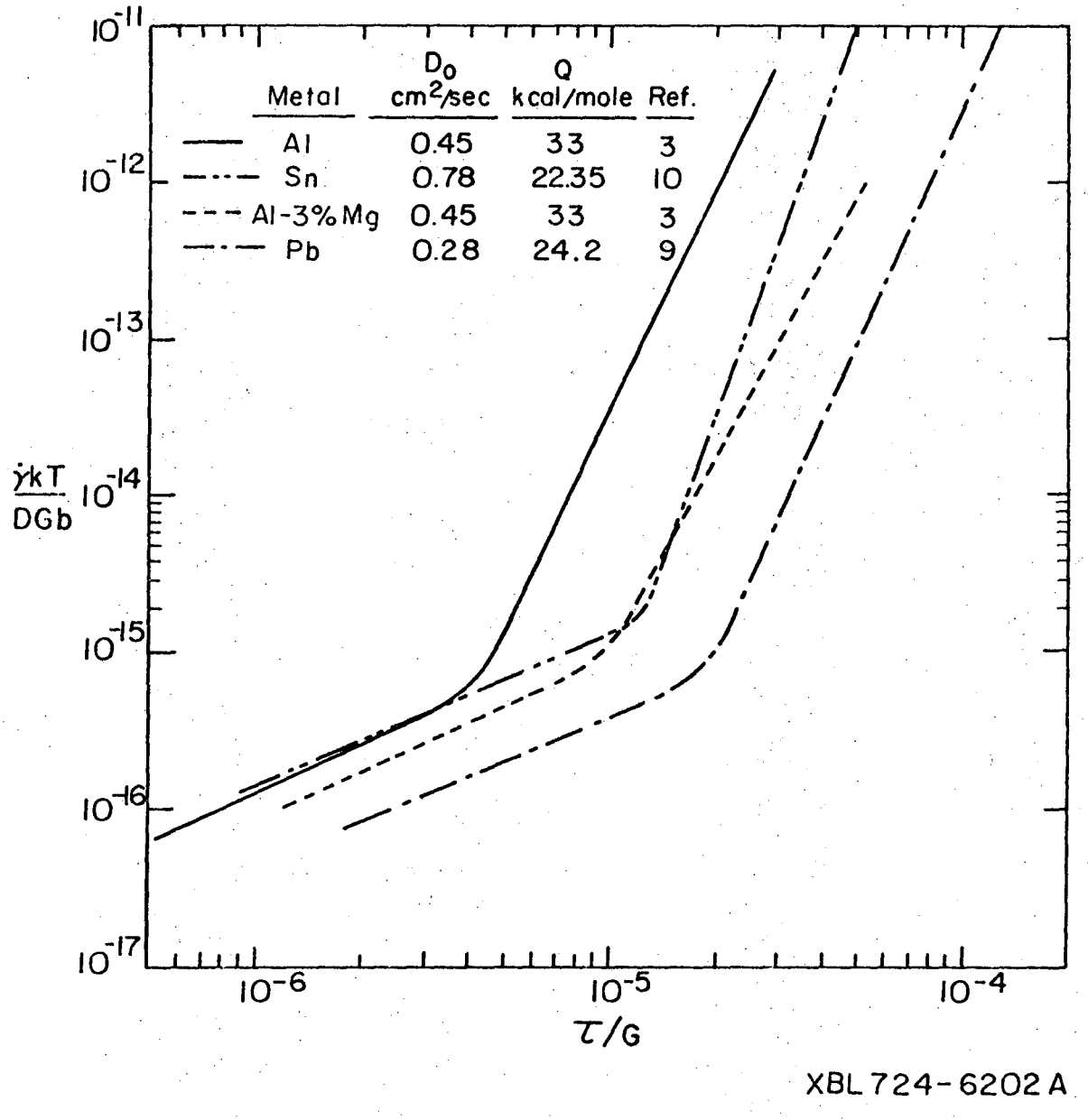

10^{-10}
10^{-11}
10^{-12}
10^{-13}
10^{-14}
10^{-15}
10^{-16}
10^{-17}
$\frac{\dot{\gamma} kT}{DGb}$
o Lead
Δ Tin
I
I
II
II
10^{-6}
10^{-5}
10^{-4}
τ/G
XBL724-6180A

Fig. 5


Metal
D_o cm²/sec
Q kcal/mole
Ref.
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Sn 0.78 22.35 10
Al-3%Mg 0.45 33 3
Pb 0.28 24.2 9
10^{-11}
10^{-12}
10^{-13}
10^{-14}
10^{-15}
10^{-16}
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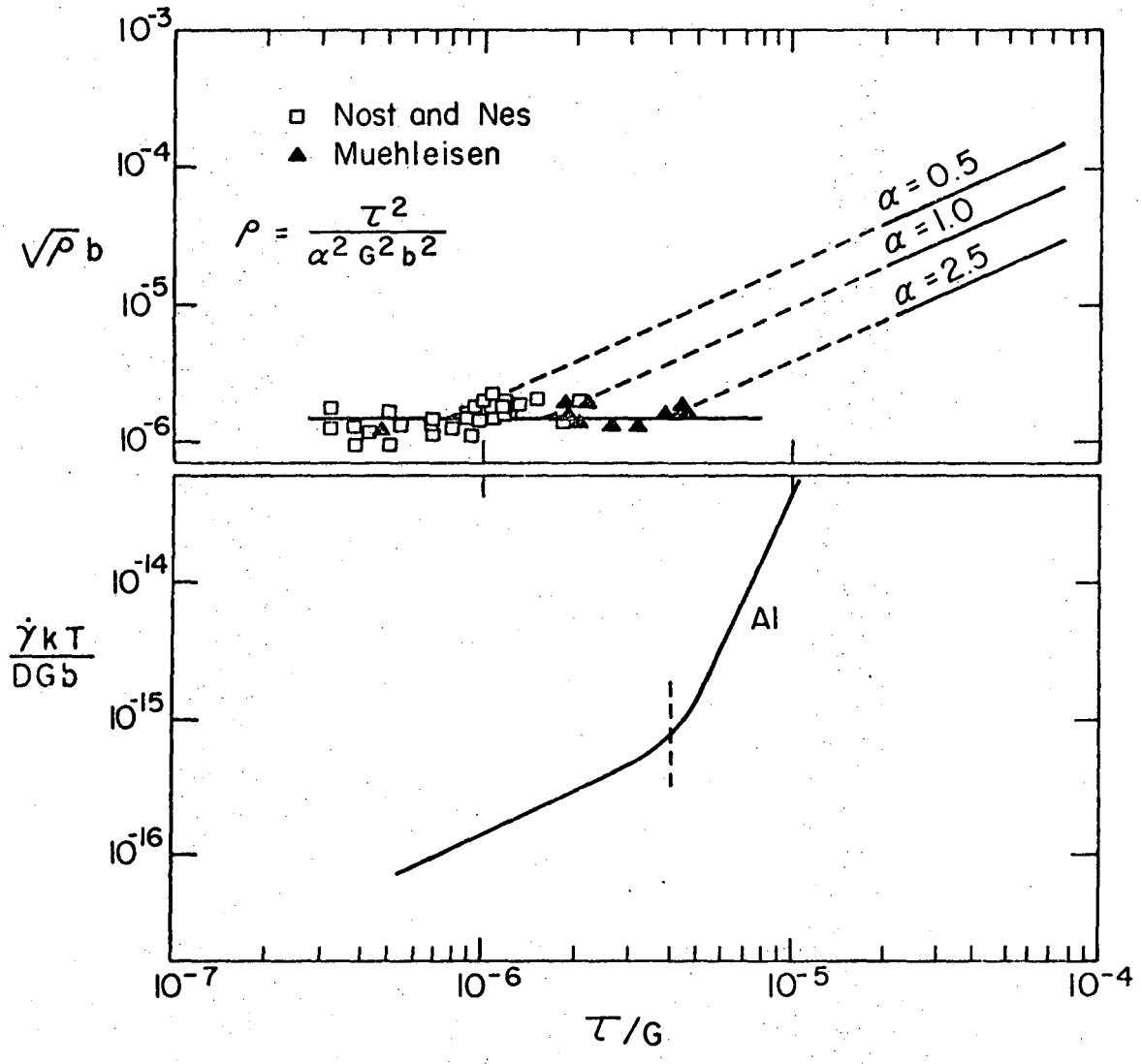
τ/G
XBL 724-6202 A

Fig. 6



XBL 724-6204A

Fig. 7

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