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Authors

Grivas, D.
Marty, K.L.
Morris, J.W.

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STEADY-STATE CREEP OF A LEAD-TIN EUTECTIC ALLOY

BY

D. Grivas, K. L. Murty*, and J. W. Morris, Jr.
 Department of Materials Science and Engineering,
 University of California, Berkeley and
 Center for the Design of Alloys,
 Inorganic Materials Research Division,
 Lawrence Berkeley Laboratory

ABSTRACT

Steady-state creep rates of lead-tin eutectic alloy (Pb-62Sn) samples of grain size from 5.5 to 9.9 μm were measured over a range of temperatures from 0° to 160°C and strain rates from 2.66×10^{-5} to 1.33×10^{-2} sec.^{-1} . The alloy exhibited both conventional and superplastic creep over this range of experimental variables. The data were consistent with the assumption that the two creep behaviors occur simultaneously via independent mechanisms. The data may be represented by the dimensionless constitutive relation

$$\dot{\epsilon}^* = 900 (\dot{\epsilon}^*)^2 (d^*)^{-1.8} \exp(-11,500/RT) + (1.3 \cdot 10^{15}) (\dot{\epsilon}^*)^{7.1} \exp(-19,400/RT)$$

where the first term on the right gives the rate of superplastic creep and the second term gives the rate of conventional creep.

*Present address: University of Newcastle
 Department of Metallurgy
 New South Wales
 2308 Australia

1. Introduction

A central problem in the study of high temperature creep is the formulation of reliable constitutive equations to represent the creep data. While a precise constitutive equation cannot be written down until the mechanisms of creep are fully understood, the semi-empirical relations proposed by Dorn and his associates have been shown to represent the steady-state creep behavior of a number of important systems. The relevant work is summarized in a recent review by Bird, Mukherjee, and Dorn⁽¹⁾, who offer a general equation which we may write in the dimensionless form:

$$(\dot{\gamma}^*) = A(\tau^*)^n (d^*)^{-m} \exp(-\Delta H^*/RT) \quad (1)$$

The dimensionless shear strain rate ($\dot{\gamma}^*$) appearing in this equation is defined in the Appendix, as are the dimensionless shear stress (τ^*) and grain size (d^*); ΔH^* is an apparent activation energy for creep; the symbols A, m, and n represent constants. Most of the suggested constitutive equations for high temperature creep can be drawn from equation (1) through proper choice of the constants A, m, and n.

Bird, et al⁽¹⁾ support equation (1) with an extensive compilation of data on the steady-state creep of metals and simple alloys. They further conclude, on the basis of limited data, that equation (1) will also represent the steady state creep of materials which exhibit superplasticity. They specifically suggest that as τ^* is decreased a superplastic material will pass through three distinct regions of steady state creep behavior, in each of which a particular variant of equation (1) will be obeyed. (1) When τ^* is sufficiently large they predict a conventional steady state creep controlled by a dislocation climb mechanism. They

hence anticipate a stress exponent (n) in the range 4-7, an apparent activation energy (ΔH^*) near that for bulk diffusion, and a creep rate independent of grain size ($m=0$). (2) For smaller τ^* they suggests a region of well-defined "superplastic" creep. On semi-empirical grounds they anticipate $m=2$, ΔH^* near that for grain boundary diffusion, and an inverse square grain size dependence ($m=2$). (3) For very small τ^* they predict a behavior dominated by the Coble⁽²⁾ mechanism of creep through grain boundary diffusion. They hence anticipate an exponent $n=1$, an activation energy ΔH^* equal to that for grain boundary diffusion, and an inverse cube dependence on grain size ($m=3$).

Bird, Mukherjee, and Dorn support their interpretation of creep in superplastic materials by replotting the data of Avery and Backofen⁽³⁾ on a Pb-Sn eutectic alloy and the data of Ball and Hutchison⁽⁴⁾ on a Zn-Al eutectoid. They argue that both alloys show clear regions of "conventional" and "superplastic" creep, and find some evidence for Coble creep in Zn-Al at low stress. However, the data obtained in these two investigations are limited, and do not permit a full test of the proposed constitutive equations. Recently both Vaidya, Murty, and Dorn⁽⁵⁾ and Misro and Mukherjee⁽⁶⁾ have reinvestigated the steady-state creep of Zn-Al eutectoid. Both sets of investigators claim verification of the Bird-Mukherjee-Dorn representation. There is, however, no comparable data for the Pb-Sn eutectic. While several investigators^(3,7-9) have studied the steady-state creep of this alloy, no single investigation contains enough information to evaluate all of the parameters in equation (1). A superposition

of data would be of dubious value since the results of these investigations are inconsistent with one another.

We have hence undertaken a new study of the steady-state creep of the Pb-Sn eutectic alloy. In the present paper we report results obtained at intermediate and high values of the applied stress and show that the data are reasonably represented by suitable forms of the equations proposed by Bird, Mukherjee, and Dorn for "superplastic" and "conventional" creep behavior.

2. Experimental Procedure

Samples of the Pb-Sn eutectic alloy were cast from pure lead and pure tin (both 99.999) into an ingot of diameter 1". The ingot was then rolled into 7/8" diameter rods at room temperature. Specimens of the "double shear" type⁽¹⁰⁾ were machined from these rods. The specimens were annealed at 175°C to obtain different mean grain sizes, which were measured from optical or scanning electron micrographs using the mean intercept method⁽¹¹⁾. Specimens exhibited grain sizes of $7.8 \pm .4 \mu\text{m}$ after 1 day anneal, $9.9 \pm .2 \mu\text{m}$ after 10 days anneal, and $5.5 \pm \mu\text{m}$ after an anneal of 21 hours.

The creep rates of these specimens were then determined at strain rates from $2.66 \cdot 10^{-5} \text{ sec}^{-1}$ to $1.33 \cdot 10^{-2} \text{ sec}^{-1}$ and temperature from $\theta^{\circ} = 160^{\circ}\text{C}$ in an Instron testing machine. The low temperature tests were conducted in ice water, the room temperature tests in air, and the high temperature tests in an electrically heated oil bath. The bath temperatures were monitored with chromel-alumel thermocouples and were maintained constant to $\pm 1^{\circ}\text{C}$.

3. Results

Apparent steady state creep was easily obtained at all temperatures and strain rates used in this series of tests. A logarithmic plot of the measured steady state strain rate as a function of the applied stress appears as in figure 1, which shows the data for the specimens of mean grain size ~ 9.9 μ m. The curve divides naturally into two distinct regions: a high stress region with slope ~ 7 and a lower stress region with slope ~ 2 . These regions of creep behavior seem associated with different dominant creep mechanisms. Samples tested in the high stress region show significant primary creep, while samples tested in the lower stress region do not; samples tested in the lower stress region show creep rates which depend markedly on grain size, while samples tested in the high stress region do not. Hence, assuming that equation (1) applies, we must anticipate that the two distinct regions of creep behavior will be governed by different variants of the equation. In the following we refer to the two types of creep behavior as "conventional" and "superplastic" creep.

(a) The conventional creep region.

The steady state creep data in the high stress region showed no evidence of dependence on grain size. Hence, the exponent m must be set equal to zero in equation (1), leaving three independent parameters: the activation energy, ΔH^* , the stress exponent, n , and the coefficient A . These parameters should be determined independently. However, given the large value of the stress exponent ($n \sim 7$) in conventional creep, we were unable to obtain enough data points at fixed $\dot{\epsilon}^*$ to permit an independent determination of ΔH^* . We therefore found values of n and ΔH^* through a

consistency procedure. Approximating $n = 7$, we approximated ΔH^* using data taken at fixed strain rate. We then assumed this value of the activation energy and adjusted n to achieve the best representation of the data with an equation of the form (1).

Specifically, if we assume $n = 7$, fix the strain rate, $\dot{\gamma}$, and measure the required shear stress τ as a function of absolute temperature T , then it follows from equation (1) and the definitions of $\dot{\gamma}^*$ and τ^* given in the Appendix that ΔH^* is simply related to the slope of a plot of $\ln \tau^7 / (G^6 T)$ vs. $1/T$. Such a plot is shown in figure 2. The data obtained with samples of all three grain sizes essentially coalesce. The slope of a least-squares line faired through this data gives $\Delta H^* = 19.4 \pm .4 \text{ kcal/mole}$ as an estimate of the activation energy for creep. This activation energy is approximately 3 kcal/mole less than that for creep in pure tin and pure lead (both have $\Delta H^* \sim 23 \text{ kcal/mole}$)⁽¹²⁾, and is less than the activation energy for self-diffusion by about 5 kcal/mole for pure lead and 4 kcal/mole for pure tin⁽¹³⁾.

Given the activation energy ΔH^* , if equation (1) is valid the data will coalesce onto a straight line in a plot of $\ln \dot{\gamma}^* \exp(\Delta H^*/kT)$ against $\ln(\tau^*)$. The line will have slope n and intercept A . Such a plot is shown in figure 3. The data do essentially coalesce. A least squares line then yields $n = 7.1 \pm .3$ and $A = 1.3 \cdot 10^{15}$. These values are comparable to those obtained by Mohamed, et al⁽¹²⁾ for creep of tin over roughly the same range of τ^* ; $n = 6.5$ and $A = 3.6 \cdot 10^{15}$.

Hence, as demonstrated in figure 3, conventional creep of the lead-tin eutectic alloy may be represented by an equation of the form (1):

$$\dot{\gamma}^* = 1.3 \times 10^{15} (\tau^*)^{7.1} \exp(-19400/RT) \quad (2)$$

The rate-controlling step in conventional creep is generally believed to be the stress-assisted climb of dislocations in the bulk metal, though specific details of the mechanism remain in doubt. (1)

(b) The superplastic region

At lower stress $n = 2$, and hence the Pb-Sn eutectic satisfies the conditions for superplastic creep (13). Creep behavior in the superplastic region shows evident grain size dependence. Assuming a constitutive equation of the form (1), the coefficient A and the exponents n , m , and ΔH^* must be determined.

Assuming equation (1), an independent determination of the activation energy ΔH^* may be made from the slope of a plot of $\ln \dot{\gamma}T$ against $1/T$ at constant applied stress and grain size. Plots for each of the grain sizes used in this research are shown in figure 4. Least-squares lines through these points yield the data appearing in table I., and gives an average value of ΔH^* of 11.5 kcal/mole. This value is identical to that reported by Bandolet and Suery (7) by Cline and Alden (8). The activation energy for creep is about 0.5 of that for self-diffusion in Sn (12), and hence is of the right order of magnitude to be interpreted as an activation energy for grain boundary diffusion, as suggested by Bird, Mukherjee, and Dorn (1).

One should, however, note that the data plotted in figure 4 show an apparently consistent curvature, which indicates either that ΔH^* is a slightly increasing function of temperature, or that it depends on some

other experimental variable which is a function of temperature. The latter point of view is more consistent with available data. The values of ΔH^* obtained by Bandedet and Suery⁽⁷⁾ and by Cline and Alden⁽⁸⁾ are identical to those we found, but were taken over different temperature ranges: -44°C to 19°C in the work of Bandedet and Suery⁽⁷⁾, and 0° to 80°C in the work of Cline and Alden⁽⁸⁾. While previous work on the Pb-Sn eutectic has not been reported in sufficient detail to compare this observation, a similar increase of the activation energy with temperature is apparent in the data of Ball and Hutchison⁽⁴⁾ and of Vaidya, Murty, and Dorn⁽⁵⁾ on the superplastic creep of the Zn-Al eutectoid. It was not possible to study the temperature dependence of ΔH^* in detail in the present work. The reported value should be regarded as an average over a range of experimental temperatures.

Given a value for the activation energy, ΔH^* , equation (1) predicts that the data for superplastic creep of samples of given grain size will coalesce into a straight line of slope n in a plot of $(\dot{\tau}^* \exp(\Delta H^*/RT))$ against (τ^*) . This coalescence is illustrated in figures 5 and 6. In figure 5 we have plotted the steady-state creep data obtained with samples of $d=5.5 \mu$ and $d=7.8 \mu$ at various test temperatures. The plot shows that, in the superplastic region, the data nearly superimpose on a line of slope close to 2.0 for each grain size. The transition to the conventional creep mechanism is also apparent. The separation of the creep curves at higher stress is due to the approximate doubling of the apparent activation energy for creep which occurs when superplastic creep is superceded by conventional creep. In figure 6 we compare the super-

plastic data obtained with samples of $d=9.9\mu\text{m}$ with the curves shown in figure 5. The data for the larger grain size may also be represented by a straight line of slope $n \approx 2$ in the superplastic region. A least squares analysis of the data for each grain size in the superplastic region revealed the results shown in Table I. From these values an average slope $n=1.95 \pm .23$ was obtained, in essential agreement with the estimate $n=2$. From Table I it can be noticed that there is no apparent dependence of n and ΔH^* on the grain size.

The grain size dependence of superplastic creep is apparent in figure 6. Given equation (1) the value of the exponent m can be computed in either of two ways: (1) a plot of the logarithm of the temperature-compensated strain rate ($\dot{\gamma}^* \exp(\Delta H^*/RT)$) against the logarithm of the mean grain diameter (d^*) at constant stress should yield a straight line of slope (m), while (2) a plot of $\ln(\dot{\gamma}^*/G)$ vs. $\ln(d^*)$ at a constant value of the temperature compensated strain rate should yield a straight line of slope (m/n). The plots are shown in figure 7. Both yield $m=1.8$, in rough agreement with the suggested value $m = 2$.

Finally, given equation (1), a logarithmic plot of the strain rate compensated for temperature and grain size, $\dot{\gamma}^*(d^*)^m \exp(\Delta H^*/RT)$, against τ^* should yield a straight line of slope n and intercept A . The plot is shown in figure 8. The value obtained for A is 900, and for n is $1.97 \pm .03$.

We hence concluded that the superplastic creep of these samples of Pb-Sn eutectic may be represented by a constitutive equation of the form (1):

$$\dot{\gamma}^* = 900 (\tau^*)^2 (d^*)^{-1.8} \exp(-11.5/RT) \quad (3)$$

The theoretical basis for equation (3), the rate-controlling step in superplastic creep, remains cloudy. Reasoning from data on superplasticity in zinc-aluminum alloys similar to the data reported here, Ball and Huchison⁽⁴⁾ suggested a model based on the dissolution of dislocation pile-ups through dislocation climb in grain boundaries. While this model yields a constitutive equation similar to equation (3), it has been questioned on empirical grounds by Nicholson⁽¹⁴⁾. The other models known to us lead to constitutive equations which do not agree with equation (3).

(3) Synthesis

We have shown above that the high temperature steady-state creep behavior of the lead-tin eutectic alloy at relatively high stress may be represented by equation (2), while the behavior at lower stress obeys equation (3). The values of the parameters appearing in these equations are roughly in agreement with those empirically suggested by Bird, Mukherjee, and Dorn⁽¹⁾. In their viewpoint these equations represent simultaneous, independent creep mechanisms. A complete constitutive equation for steady-state creep of the lead-tin eutectic should then be obtained by summing equations (2) and (3):

$$\dot{\gamma}^* = 900 (\tau^*)^2 (d^*)^{-1.8} \exp(-11.500/RT) + (1.3 \times 10^{15}) (\tau^*)^{7.1} \exp(-19.400/RT) \quad (4)$$

The results of several independent creep experiments are compared with

the prediction of equation (4) in figure 9. Although the fit of the 5.5 μm and 9.9 $^{\circ}\text{C}$ seems to be quite good, the 7.8 μm grain size deviates from the above equation in the superplastic region by a factor of 1.8. This is most likely due to an error in the grain size determination. The fit is very good in the conventional creep region where there is no grain size dependence. If m is taken as 2 then A becomes 209, and an equally good fit can be observed. The shape of the computed curves illustrates that the value of the right hand side of equation (4) is completely dominated by the larger of its two terms except over a narrow region of (τ^*) where the two terms have nearly the same value.

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APPENDIX

The quantities appearing in equation (1) are defined as follows:

$$\dot{\gamma}^* = \dot{\gamma} kT / D_0 G b \text{ (a dimensionless strain rate).}$$

$$\tau^* = \tau / G \text{ (a dimensionless shear stress).}$$

$$d^* = d / b \text{ (a dimensionless grain size).}$$

The symbols have the following meanings:

$\dot{\gamma}$ = strain rate.

k = Boltzman's constant.

T = absolute temperature.

D_0 = a characteristic diffusivity, chosen equal to the pre-exponential D_0 in the diffusion equation for pure Sn, ($.08 \text{ cm}^2 / \text{sec}$)⁽¹⁵⁾.

G = a characteristic stress, taken equal to the shear modulus of pure Sn, ($2 \times 10^{11} \text{ (d/cm)}$)⁽¹⁵⁾.

b = a characteristic length, taken equal to the Burger's vector of pure Sn, (3.18 (A))⁽¹⁵⁾.

τ = resolved shear stress.

d = mean grain diameter.

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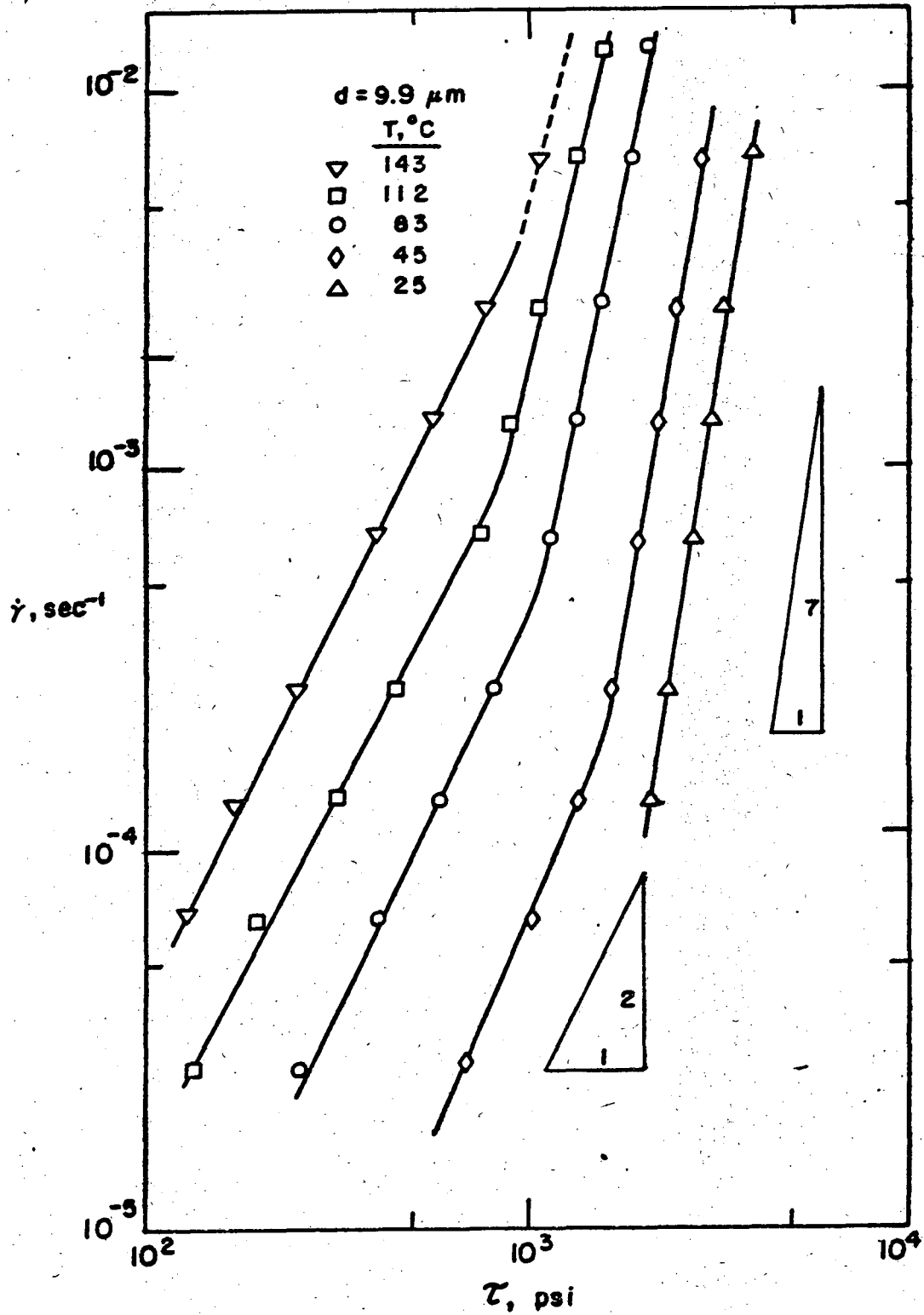
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TABLE I

<u>Grain Size</u>	<u>n Superplastic</u>	<u>ΔH^* superplastic</u>
5.5 μm	2.06 \pm 0.1	11.45 \pm 0.11 kcal/mole
7.8	1.76 \pm 0.12	11.83 \pm 0.46
9.9	2.05 \pm 0.06	11.15 \pm 0.25

FIGURE CAPTIONS

- Fig. 1. Stress vs. steady state creep rate of Pb-Sn eutectic at various temperatures for the 9.9 μm grain size, indicating the two distinct regions at different stress levels.
- Fig. 2. Arrhenius plot of $\ln(\tau^7/G^6T)$ vs. $1000/T$ for the computation of the activation energy in the conventional creep region.
- Fig. 3 Plot of $\dot{\gamma}^* \exp(\Delta H^*/RT)$ vs τ^* for the conventional creep region revealing no grain size dependence and a slope $n = 7.1$.
- Fig. 4. Arrhenius plot of $\ln \dot{\gamma}T$ for the computation of the activation energy for superplastic creep vs $1000/T$ ($\dot{\gamma}T$ is related to τ^* by a constant as can be seen from the definitions in the appendix).
- Fig. 5. The logarithmic plot of the temperature compensated strain rate vs τ^* for the 5.5 μm and 7.8 μm grain size.
- Fig. 6. The logarithmic plot of the temperature compensated strain rate vs τ^* for the 9.9 μm including the superplastic region of the 5.5 and 7.8 μm grain size.
- Fig. 7. The effect of grain size on the strain rate and stress in the superplastic region.
- Fig. 8. Plot of the strain rate compensated for temperature and grain size vs τ^* for all superplastic data.
- Fig. 9. A plot of τ^* vs $\dot{\gamma}^*$ as obtained experimentally and from equation (4).



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Fig. 1

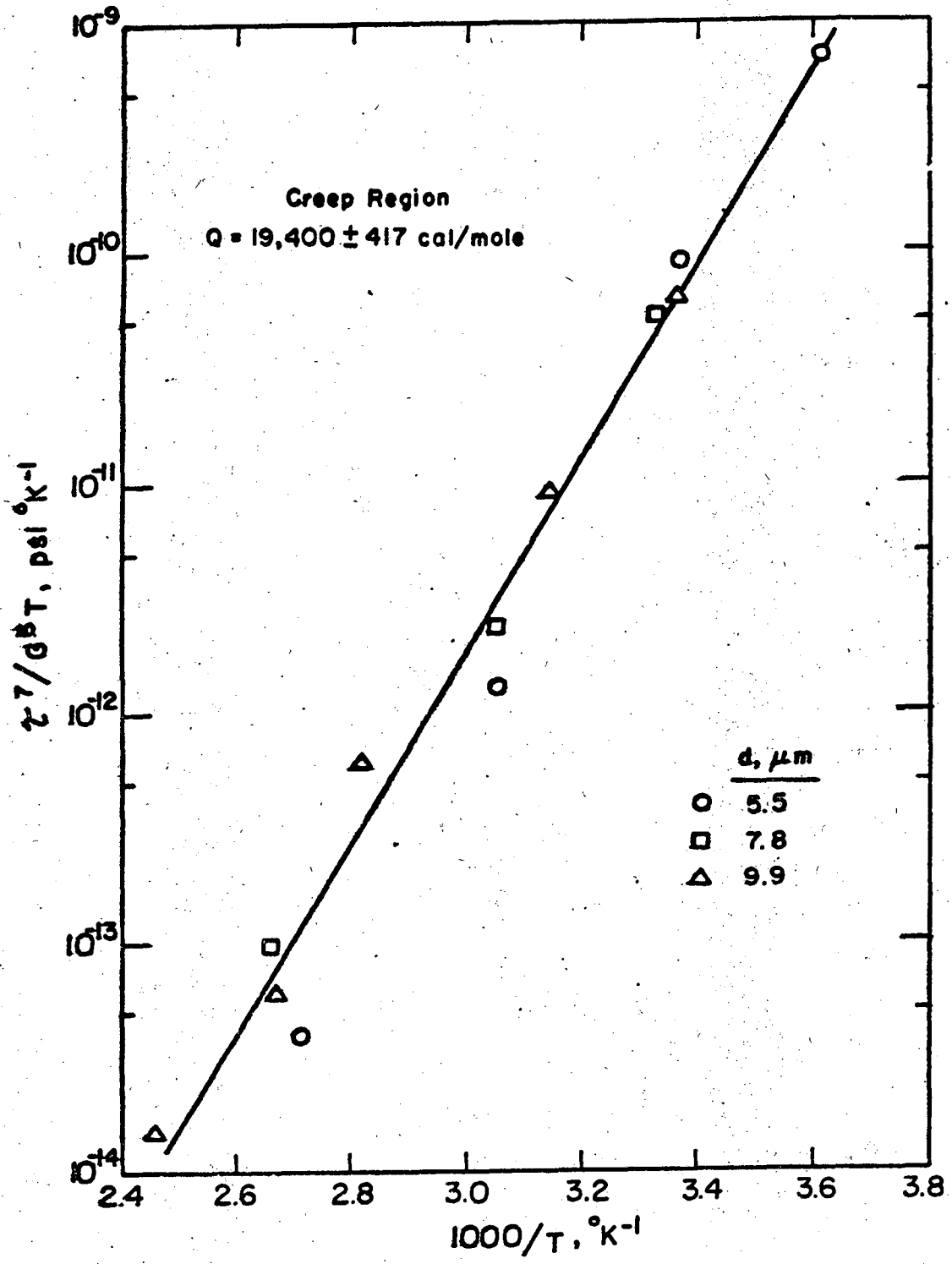
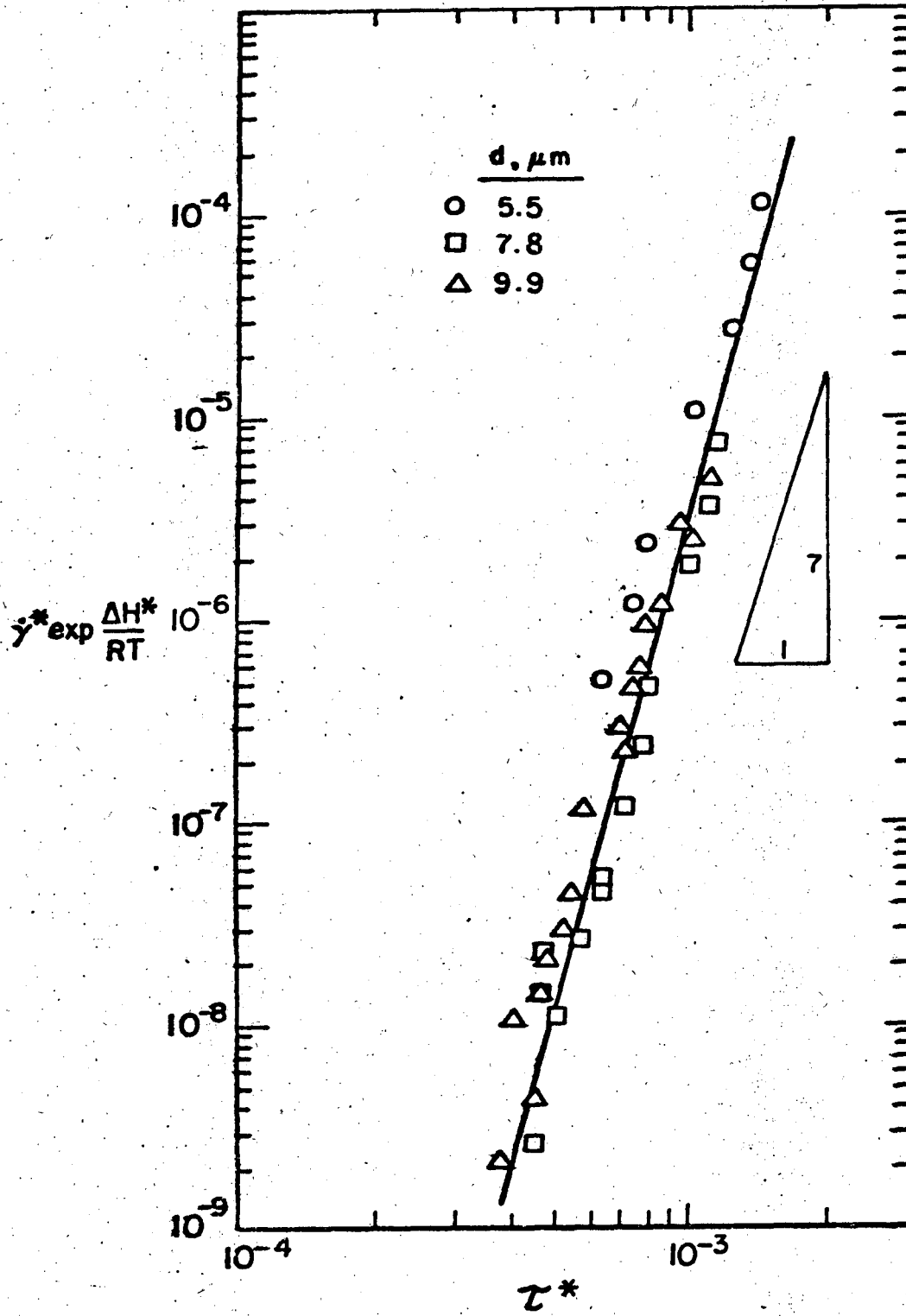
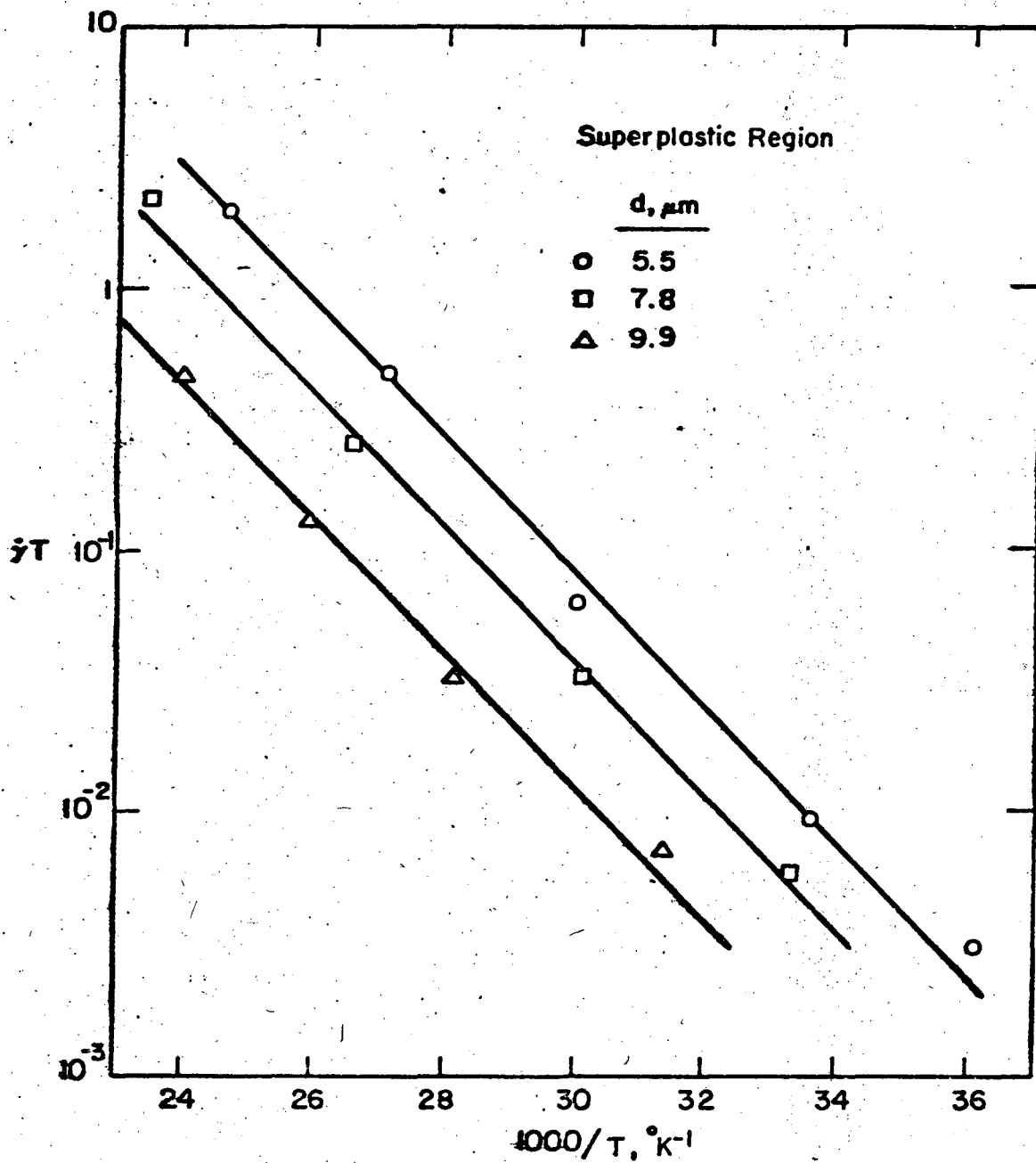


Fig. 2



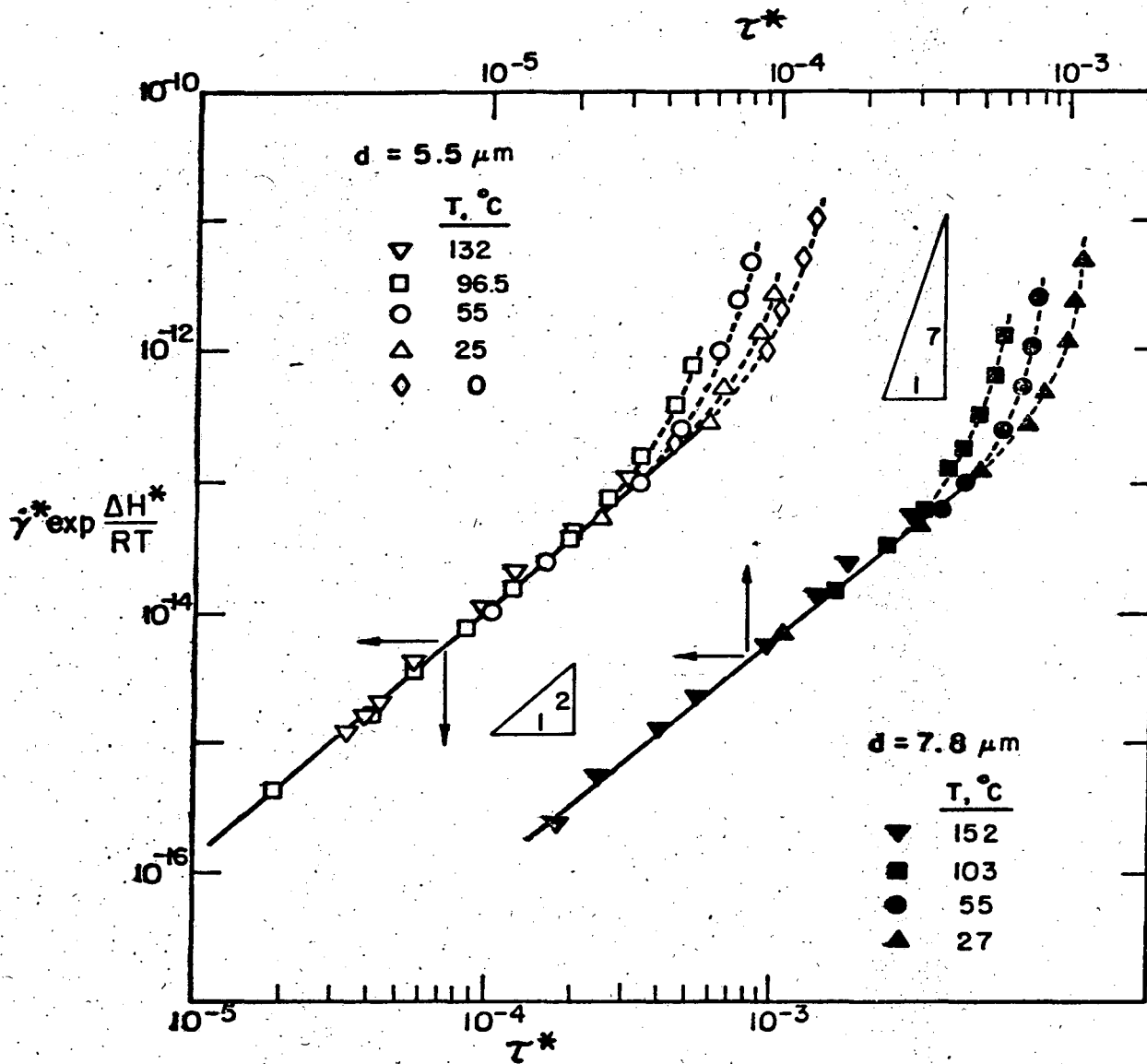
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Fig. 3



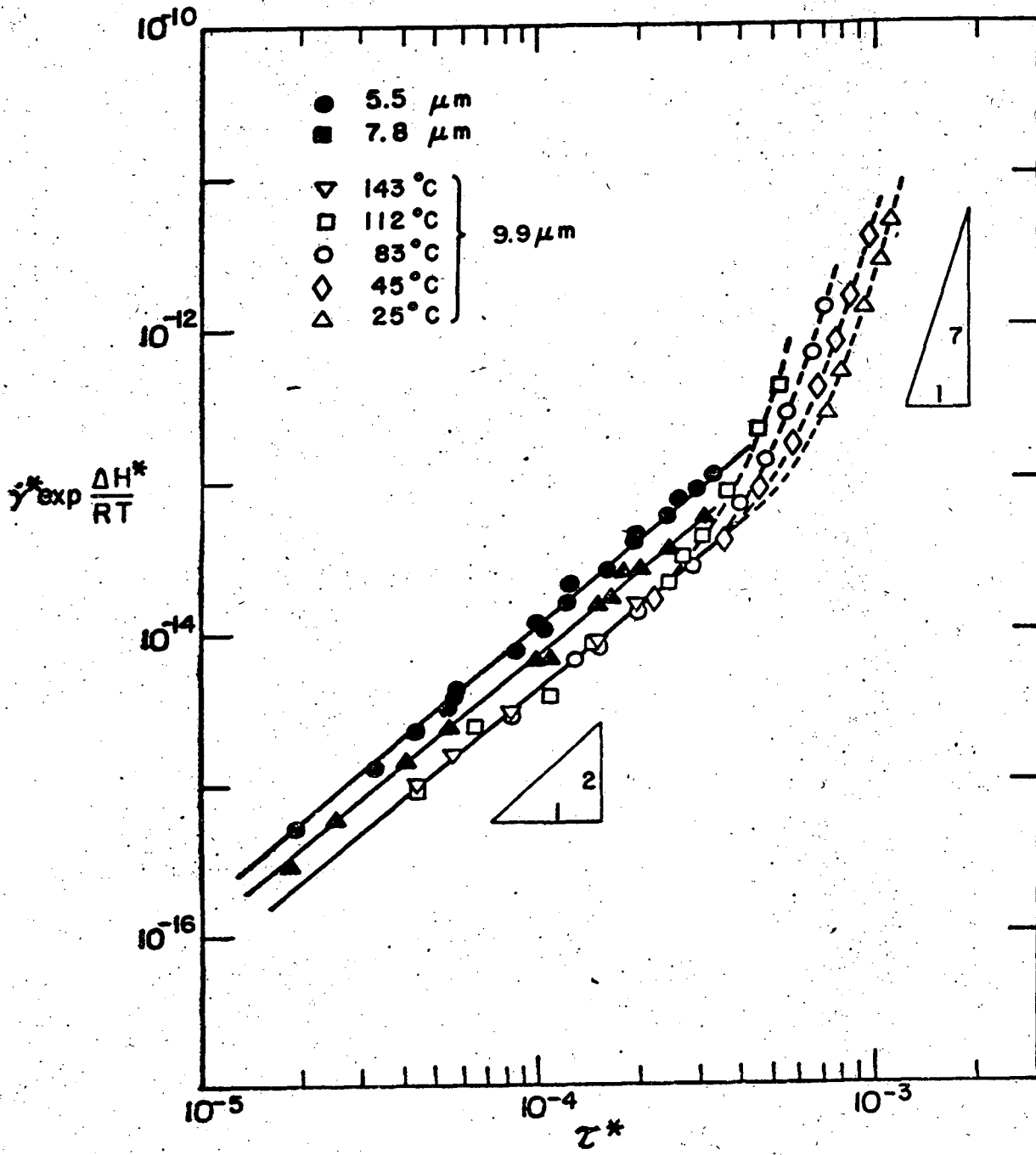
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Fig. 4



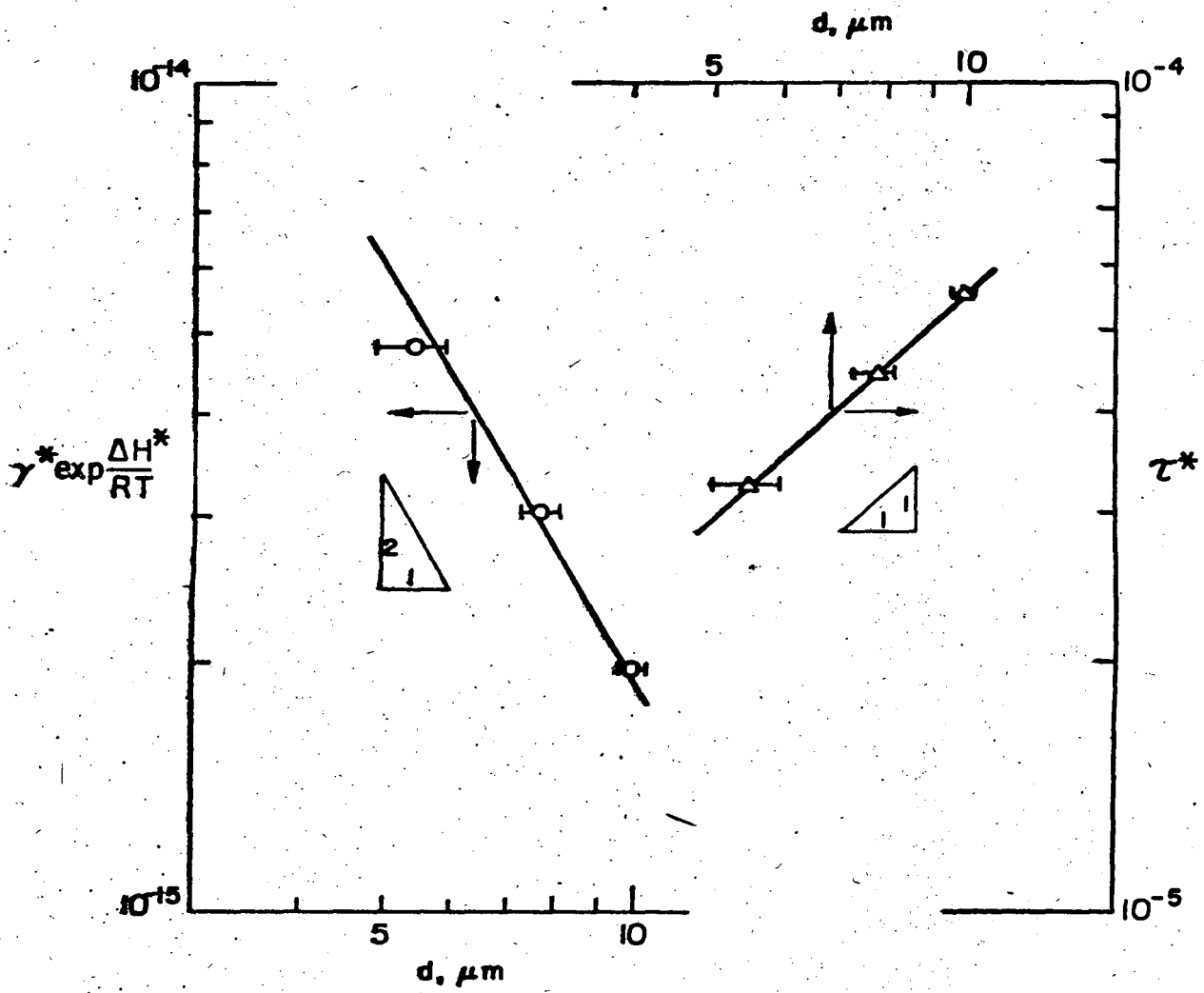
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Fig. 5



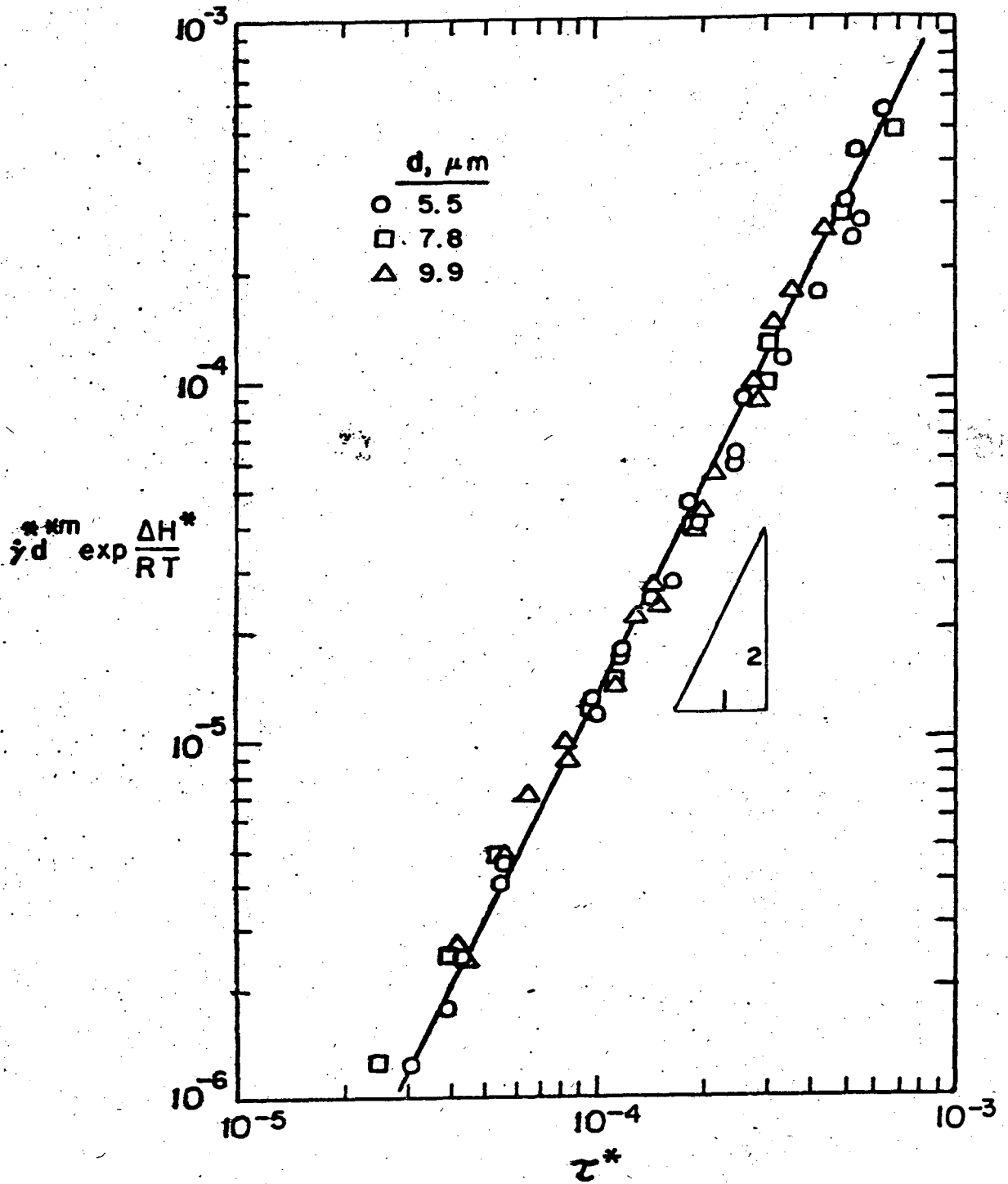
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Fig. 6



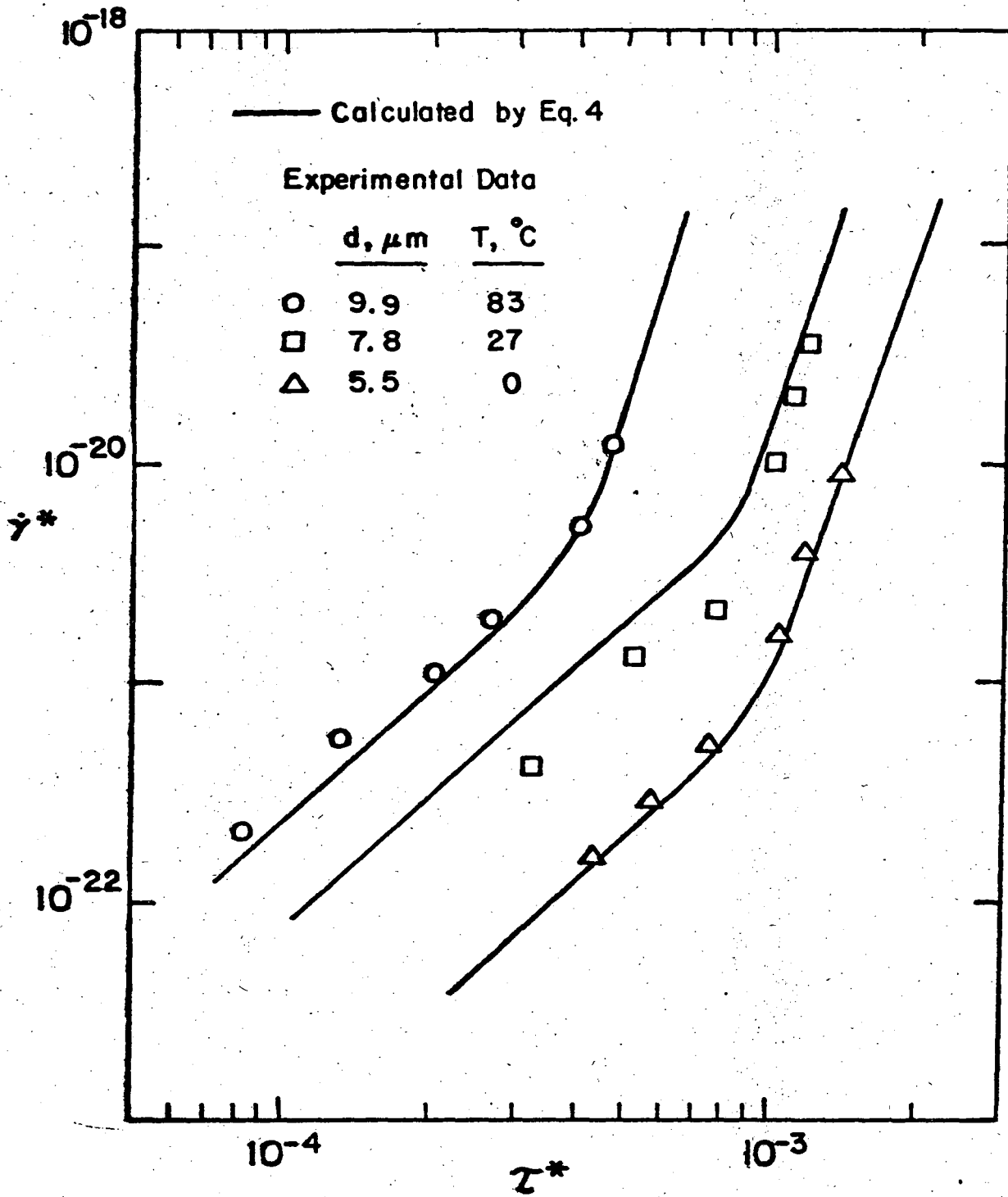
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Fig. 7



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Fig. 8



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Fig. 9

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