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Indentation creep of lead and lead–copper alloys

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Stress exponent values have been determined in Pb and Pb–Cu alloys with small Sn, Se and Pd additions by indentation methods (long time hardness tests) to evaluate their applicability as compared with tensile tests. Homogeneous, fine grained alloys were obtained by induction melting and thermo-mechanical treatments. Grain size was 38–60 μm in alloys and 183 μm in pure lead. Stress exponent values, i.e. of 11–12 agree between different methods of derivation and, in fine grained material, with tensile methods. The largest differences in pure lead, i.e. 10–11 versus 7–8 are attributed to high strain rates when indentation size is comparable to grain size. In all cases indentation and tensile tests indicate the same deformation mechanism, namely slip creep. The indentation test is thus considered useful, within limits, to acquire information on the deformation mechanism.

1. Introduction

Several papers have addressed the possibility of gaining information on creep properties by the use of indentation or impression tests, e.g. long time hardness tests [1–4]. A constant load is applied on the surface of the sample with a suitable indentor for a period which largely exceeds the duration of a standard hardness test (i.e. up to 360 min). The variation of the indentation size, expressed as diameter (Brinell test), diagonal length (Vickers test), or penetration depth of a cylindrical indentor (impression test), is followed with time. The first part of the curve records an increase in the concerned variable with time, with decreasing rate, followed by a stationary region where size increases linearly with time. On such a test it is obviously not possible to record a third stage of the curve, as opposed to what happens in an ordinary creep test [1, 5].

These tests are simpler than creep tests because they do not require sample machining and can be carried out on small, simple flat specimens. Moreover, the effect of temperature, provided it does not give rise to microstructural changes, can be evaluated on a single specimen [5–7].

The term "impression creep" was first suggested by Chu and Li [8]. By using a cylindrical indentor, they claimed to avoid the difficulty of figuring out the strain rate for a pyramidal or spherical indentor, and attributed the significance of constant strain rate under constant stress to steady state penetrating velocity under constant indenting load. They determined, through finite element analysis, that, in an impression test with a cylindrical indentor, the strain rate, ε', equals the relation between penetration velocity (calculated from the penetration depth versus time plot) and indentor diameter. Also, they determined that the stress equals 1/3 of the stress below the indentor flat end, and they obtained the stress exponent, n.

Most of the relevant work in the area of impression and indentation creep has been carried out on lead alloys, where indentation methods have generally been preferred, although in some cases more than one method has been tested on the same material. Juhász *et al.* [9] carried out tests on a superplastic lead–tin alloy using a Vickers microhardness tester and defined an m coefficient which would be the reciprocal of n, the stress exponent in steady state creep, as: $m = (\partial \ln H_V/\partial \ln d')_d$, where H_V is the Vickers microhardness number and d' the rate of variation of the indentation diagonal length, d. As the power law which rules creep processes is of the type $\varepsilon' = B\sigma^n$, where ε' is the strain rate and σ is the stress, m can be defined as $1/n$, that is: $m = (\partial \ln\sigma/\partial \ln\varepsilon')_\varepsilon$. This means that they attributed the same physical significance to stress and hardness on one side (both have a force per unit area dimension) and to strain rate and rate of variation of the diagonal of the indentation on the other. They verified this relation for their experiment and obtained the same m and activation energy values from indentation and creep tests. In a different paper Juhász *et al.* reported results of impression tests on the same superplastic lead–tin alloy and obtained the same results as with the indentation method [5].

Mulhearn and Tabor determined the value of the steady state stress exponent, as well as the activation energy of the creep process, for temperatures above 0.6 T_M in pure lead, using a spherical indentor [10]. They related hardness to indentation time to calculate the value of the stress exponent through the expression $-(n + 1/2)\log H_V = \log t$ and obtained a stress exponent of 10. Carrying out tests at different temperatures, keeping load and indentor geometry

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(diameter) constant, they determined an activation energy of 117 kJ mol^{-1}, close to the activation energy for lattice self-diffusion.

Hooper and Brookes carried out indentation creep tests on 99.99% lead with Vickers, Knoop and conical indentors [2] and achieved similar results through creep and indentation tests for temperatures above 0.5 T_M. Below 0.5 T_M, at moderate loads, they observed an incubation period, which depended on applied load, temperature, crystallographic orientation and indentor geometry.

In the case of lead alloys, the simplicity of these tests, especially when carried out on conventional hardness machines, compares favourably with the difficulties in machining tensile pieces. This latter feature has made the number of creep studies on these materials scarce compared with other engineering materials. The advantage is not so clear, however, for impression tests which require a specially built machine.

From the literature data mentioned above, it is reasonably clear that impression and/or indentation tests allow us to make a good estimate of the stress exponent in superplastic lead alloys, that tests on pure lead have produced results well apart from those obtained in regular creep tests and that the relation between indentor and grain sizes plays an important role.

It has been considered useful to test the validity of these methods in the case of lead alloys with comparatively good mechanical properties, such as lead–copper alloys used in the manufacture of chemical plants, where life service depends to a large extent on fine grain size and the stability of microstructure and mechanical properties. As lead exhibits dynamical recrystallization at room temperature, this requires the use of specific alloying elements to enhance grain nucleation, prevent grain growth and retain good corrosion resistance. These requirements, as well as economical reasons, indicate lead copper alloys with small tin, selenium and palladium additions as the most adequate for chemical plant applications.

In the course of a detailed research to fully characterize Pb–Cu (Sn–Se–Pd) alloys from the microstructural and mechanical points of view, a number of results have been produced on the creep behaviour and deformation mechanisms of these alloys [11] and it has been considered useful to determine stress exponent values by indentation tests using exactly the same material, in the same conditions, to evaluate to what extent they can replace more elaborate testing procedures to gain information on creep behaviour. Because of differences between the stress exponents derived by indentation methods reported by other authors and normally accepted values from creep tests in pure lead, measurements on this material, as well as on lead–copper without further additions have been carried out.

2. Experimental procedure

2.1. Alloy and sample preparation

Alloys for these experiments were first prepared as conventionally cast Pb–Cu and Pb–Sn master alloys and inert atmosphere induction melted and cast Pb–Se and Pb–Pd master alloys. Purity of lead was 99.99%.

From these, the alloys shown in Table I were cast as 150 × 95 × 20 mm plates and compositions were chemically analysed by atomic absorption or inductively coupled plasma techniques, depending on alloying element.

To ensure a given microstructure as the initial state of the material, ingots were first homogenized for 24 h at 453 K and air cooled. They were subsequently cold rolled to a 75% reduction to destroy the cast structure, further homogenized for 12 h at 553 K and finally cold rolled again to a 60% reduction. This procedure is adequate to produce homogeneous, fine grained material, without the remnants of dendritic structure.

Grain size for this condition, for all alloys and pure lead, determined by quantitative metallography as mean intercept lengths, is shown in Table II. Measurements were carried out with an IBAS 2 image analyser.

2.2. Hardness tests

After trials with different indentors, the Vickers indentor was selected, where applied load and testing time are the only variables. In the Vickers test, a diamond pyramid with square base and 136° angle at the vertex is used and the Vickers hardness number is given by $H_V = 0.1854\, F/d^2$, where F is the applied load in N, and d the average diagonal length in mm. As this indentor guarantees a constant geometry of the indentation it should be preferred to the Brinell test, where a definite ratio $K = F/D^2$ is normally maintained between the load and the square of the indentor diameter (K is 6.125 N mm^{-2} for soft materials such as lead and, if applied load were changed to determine the stress exponent, K would also change).

The variation of the indentation diagonal with time was followed for times up to 6 h. Results are normally given as the average of three tests.

3. Results

The variation of indentation diagonal length with time under constant load of 9.8 and 29.4 N has been plotted

TABLE I Composition of the alloys % mass, balance lead

Alloy	Cu	Sn	Se	Pd
A	0.04	0.05	0.02	–
B	0.04	0.05	0.02	0.03
C	0.036	–	–	–

TABLE II Grain size of all materials

Material	Grain size (μm)
Pure lead	183
Alloy C	63
Alloy A	39
Alloy B	38

in Fig. 1. In all materials, the shape of the curve is similar to that of an ordinary creep curve, with a first stage and a stationary regime region. As the hardness test is actually a compression test, it is not possible to achieve fracture of the specimen.

In order to find values for the stress exponent, n, both methods of derivation have been used for comparison. Using the Juhász, Tasnadi and Kovacs relation, $m = (\partial \ln H_V / \partial \ln d')_d$, if the rate of variation of the diagonal is plotted against the Vickers microhardness number on a double logarithmic scale, a straight line is obtained with slope $n = 1/m$ [9]. The rate of variation of the diagonal with test time, d', has been obtained by graphic differentiation of the curves in Fig. 1. Fig. 2 shows the log–log plot of the diagonal variation rate against hardness for all materials.

Using the Mulhearn and Tabor relation, $-(n + 1/2) \log H_V = \log t$, if hardness is plotted against time on a double logarithmic scale, a straight line with slope $n + 1/2$ is obtained [10]. This has been represented in Fig. 3 for all materials and both testing loads.

The stress exponent values obtained using both methods of derivation are shown in Table III. They represent computed values from the straight line least squares fit but, as will be shown later, figures after the decimal point are not really relevant.

4. Discussion

When the stress exponent, n, is obtained through either derivation method, both produce numerical results which are in good agreement, which indicates the similarity of the derivation approach. A useful finding is that the relation proposed by Juhász *et al.*, $m = (\partial \ln H_V / \partial \ln d')_d$, which they tested for microhardness tests only, is equally applicable to hardness tests.

When the results of the indentation tests are compared with those obtained in conventional tensile tests at different strain rates, shown in Table III, however, some differences are found which are worth discussing. In alloy B, namely in the one with better mechanical properties [11], similar stress exponent values are obtained by indentation and tensile methods but in alloys A and C, a difference of around two units is obtained.

For the lead–copper system, the authors are not aware of other work using either the conventional derivation of the stress exponent by strain rate variation tensile tests or by any other application of the

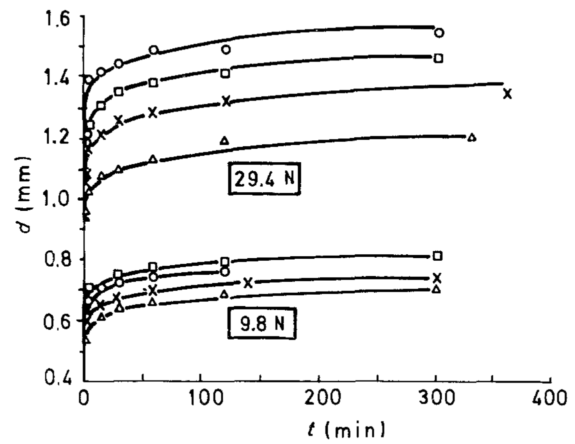

Figure 1 Variation of indentation diagonal with test time for all materials at 9.8 and 29.4 N. (□) A; (△) B; (×) C; (○) Pb.

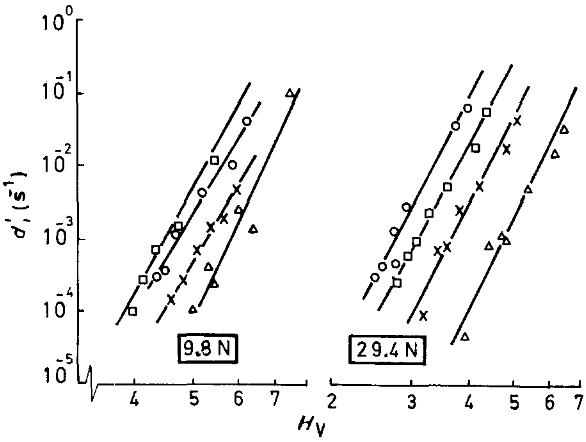

Figure 2 Derivation of the stress exponent by the Juhász method. (□) A; (△) B; (×) C; (○) Pb.

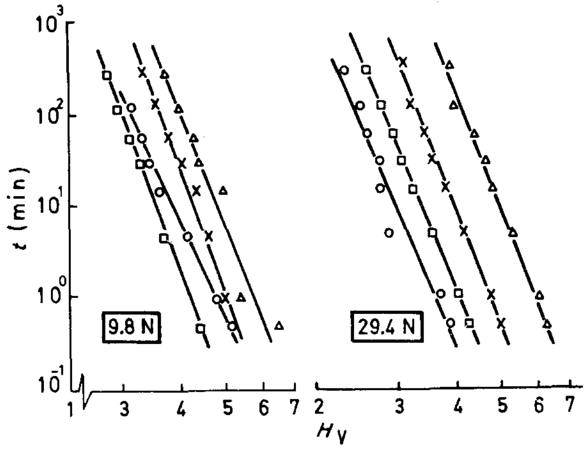

Figure 3 Derivation of the stress exponent by the Mulhearn method. (□) A; (△) B; (×) C; (○) Pb.

TABLE III Stress exponents derived from indentation and tensile tests

Material	Stress exponent			
	Indentation tests			Tensile tests
	Load (N)	Juhász	Mulhearn	
Alloy A	9.8	11.2	13.2	9.4
Alloy A	29.4	11.5	12.0	
Alloy B	9.8	13.5	12.0	12.3
Alloy B	29.4	12.4	11.5	
Alloy C	9.8	10.5	13.2	8.9
Alloy C	29.4	12.1	12.1	
Pure lead	9.8	10.5	10.3	7.4
Pure lead	29.4	12.3	11.4	

indentation or impression methods and so it is not possible to compare the present results with other work in the literature on the same system. In the case of the Pb–Sn superplastic alloy studied by Juhász *et al.*, however, larger values were also found for the stress exponent through indentation than through tensile tests, but with appreciably lower differences [9].

In the case of pure lead, the difference is of the order of four units in the stress exponent, namely 11.2 (average) by the indentation method against 7.4 by tensile methods (the latter is in agreement with the generally admitted value of 6–7). Present results, however, are similar to those by Mulhearn and Tabor, who determined, in pure lead, a stress exponent of 10 by the indentation method. They determined the variation of hardness with time of test for temperatures above 0.6 T_M only, while in our case this variation has been determined for a temperature around 0.5 T_M. In any case, when considering numerical values of the stress exponent, it should be remembered that differences in one or two units in an absolute value of 10–12 have no real physical significance when dealing with deformation mechanisms. In fact, deformation of polycrystalline materials at temperatures above $\sim 0.3\ T_M$ can take place by different deformation mechanisms [12], associated with different strain exponent value ranges. Thus, diffusional creep is linked to n values around 1, grain boundary sliding leads to n values close to 2 and mechanisms associated to dislocation movement such as slip creep are linked to n values in the 5–7 range, which moves up to 8–12 when particle reinforcement takes place. Such is the case of lead copper alloys, where different reinforcing phases are present, as shown elsewhere [13].

In any case, the better agreement that is always found for fine grained material is due to the number of grains which take part in the deformation process. When grain size is of the same order of magnitude as the size of the indentation, such as with pure lead, it is more difficult to establish an analogy between tensile tests (where a large number of grains take part in the creep process) and the present method. This can also explain the similar differences obtained by Mulhearn and Tabor, as well as the conclusion attained by Chu and Li [8], using the impression method with a cylindrical indentor on succinonitrile crystals. They obtained abnormally high strain rates when the diameter of the indentor was of the same order of magnitude as the grain size and decided not to consider those results.

With all the limitations implied in the use of indentation or, according to the previous observation, impression methods, the most interesting observation drawn from these results is that stress exponent values obtained by indentation tests and by tensile (strain rate variation) tests indicate the same deformation mechanism, attributed in our case to slip creep. This would indicate that while the similarity is only approximate, the indentation test is a simple, useful procedure, within certain limits, to acquire information on the deformation mechanism in fine grained materials with comparatively low mechanical properties.

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