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Effect of solidification conditions on deformation behaviour of semi-solid Sn–Pb alloys

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The compressive behaviour of Sn–Pb alloys is studied with materials of conventionally dendritic structure and non-dendritic (rheocast) structure obtained through mechanical stirring during solidification. The alloys are found to deform similarly in the fully solid state but their behaviour becomes very different in the semi-solid range depending on the solidification mode. The holding time in the semi-solid state before compression also affects the mechanical properties: the influence of these two parameters is discussed in terms of the initial structure of the alloy and its evolution. Advantages of using semi-solid materials in metal forming processes are also presented.

1. Introduction

Over the last few years, new metal forming processes in the semi-solid state have been developed: these processes are made possible because of the particular thixotropic properties of non-eutectic alloys when they are vigorously agitated during the early stages of solidification [1–3].

The fluid-like character of the as-produced slurry is the result of the non-dendritic structure of the primary phase and it is retained after the alloy has been fully solidified and later reheated beyond the solidus without agitation. During a compression test particularly, it was found in the case of Sn–15 wt % Pb alloy that this kind of structure can be deformed under very low stresses without any important segregation of the liquid phase except at very large strains [4].

Conversely the conventionally solidified alloy requires higher stresses and shows a completely heterogeneous structure after compression. This segregation, however, depends on the deformation rate and on the structure size, then on the cooling rate of the alloy [5].

Indeed for very fine structures, the alloy is found to behave almost similarly as the non-dendritic one, owing to the initial spheroidization of the primary solid phase during the heating time of the sample [5].

The aim of this work is to investigate more deeply the influence of the initial structure of the alloy on its compressive behaviour in the semi-solid state and to study the effect of the isothermal holding time before the compression test. Moreover, some experimental results concerning the behaviour of the alloy in the solid state at different temperatures will also be presented for comparison.

2. Experimental procedure

In this investigation, the specimen was squeezed between two parallel discs heated to the desired temperature with two 155 W cylindrical heaters. Two chromel–alumel thermocouples embedded in the bottom and the top plate were used to assure temperature control.

The compression was performed using a standard Zwick testing machine at a constant crosshead velocity of 1.67×10^{-4} m sec^{-1}. All specimens of Sn–15 wt % Pb were cylindrical, 10 mm diameter and 5 mm high, machined from as-cast ingots.

Different structures were investigated: a non-

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TABLE I Solidification conditions for the Sn–15% Pb alloys investigated, and resulting structural characteristics

Type of structure	Solidification condition	Average cooling rate $(dT/dt)_{avg}$ (°C min^{-1})	Characteristics size (μm)	Thermal treatment before testing
Non-dendritic	mechanical stirring during solidification	40	$r = 30-40$	ageing
Fine dendritic aged	in copper mould	500	$D_p = 20$	ageing
Fine dendritic	in copper mould	500	$D_p = 20$	none
Intermediate dendritic	in graphite mould	30	$D_p = 80-90$	none
Coarse dendritic	in preheated graphite mould (200° C)	8.7	$D_p = 150-160$	none
Coarse dendritic	in preheated graphite mould (400° C)	4.1	$D_p = 220-230$	none
Coarse dendritic	in a graphite mould cooled in the furnace	1	$D_p = 400-500$	none

r = radius of the primary particles.
D_p = primary dendrite arm spacing.

dendritic (rheocast) structure, made in a low temperature slurry producer at the Massachusetts Institute of Technology [2, 6] and several dendritic ones obtained by conventional solidification. For the latter, different moulding conditions were used in order to obtain different cooling rates. Table I gives the solidification conditions and the structural characteristics of the alloys investigated. One of the dendritic structures was also prepared at the Massachusetts Institute of Technology, and for this structure, as well as for the non-dendritic, the compression tests were carried out after an ageing time of about one year at room temperature. The other dendritic alloys were stored after casting in liquid nitrogen in order to avoid any evolution of the structure. Most of the tests dealing with the fine dendritic structure were, however, performed with aged specimens, but results concerning fine as-cast structures will also be presented for comparison, in order to test the influence of this ageing on the mechanical behaviour of partially remelted alloys in which the initial eutectic mixture is degenerated.

For each compression experiment, the procedure was the following: the specimen was put between the two discs preheated at the desired temperature and a heating time of 90 sec was found to be necessary for the specimen to reach this temperature. Most of the tests were, however, performed after a waiting time of 150 sec. The tests were carried out at 20, 100 and 175° C for the experiments in the solid state and 184° C (i.e. one degree above the eutectic temperature) for the semi-solid state. After all the experiments in the semi-solid state, the specimens were water-quenched to below the eutectic temperature within a few seconds. They were then sectioned, polished and etched for metallographic observations.

The size of the primary particles in the non-dendritic specimens was determined by the linear intercept method using a semi-automatic image analyser. In order to quantify the segregation of the liquid phase which takes place during compression, the fraction of the eutectic phase (and therefore the amount of liquid that was present at just above the eutectic temperature) was measured automatically on the sample cross-section after polishing and etching by use of an automatic quantitative image analyser. For the Sn–15% Pb alloy, the initial volume fraction g_{Lo} of the liquid phase at just above the eutectic temperature is 0.37 as given by the Scheil equation, the eutectic composition being at 38.1% Pb.

3. Experimental results

Fig. 1 shows optical micrographs of the different types of structures used in this investigation and listed in Table I. The non-dendritic structure consists of quasi-spherical primary particles of the tin-rich phase dispersed in the eutectic mixture.

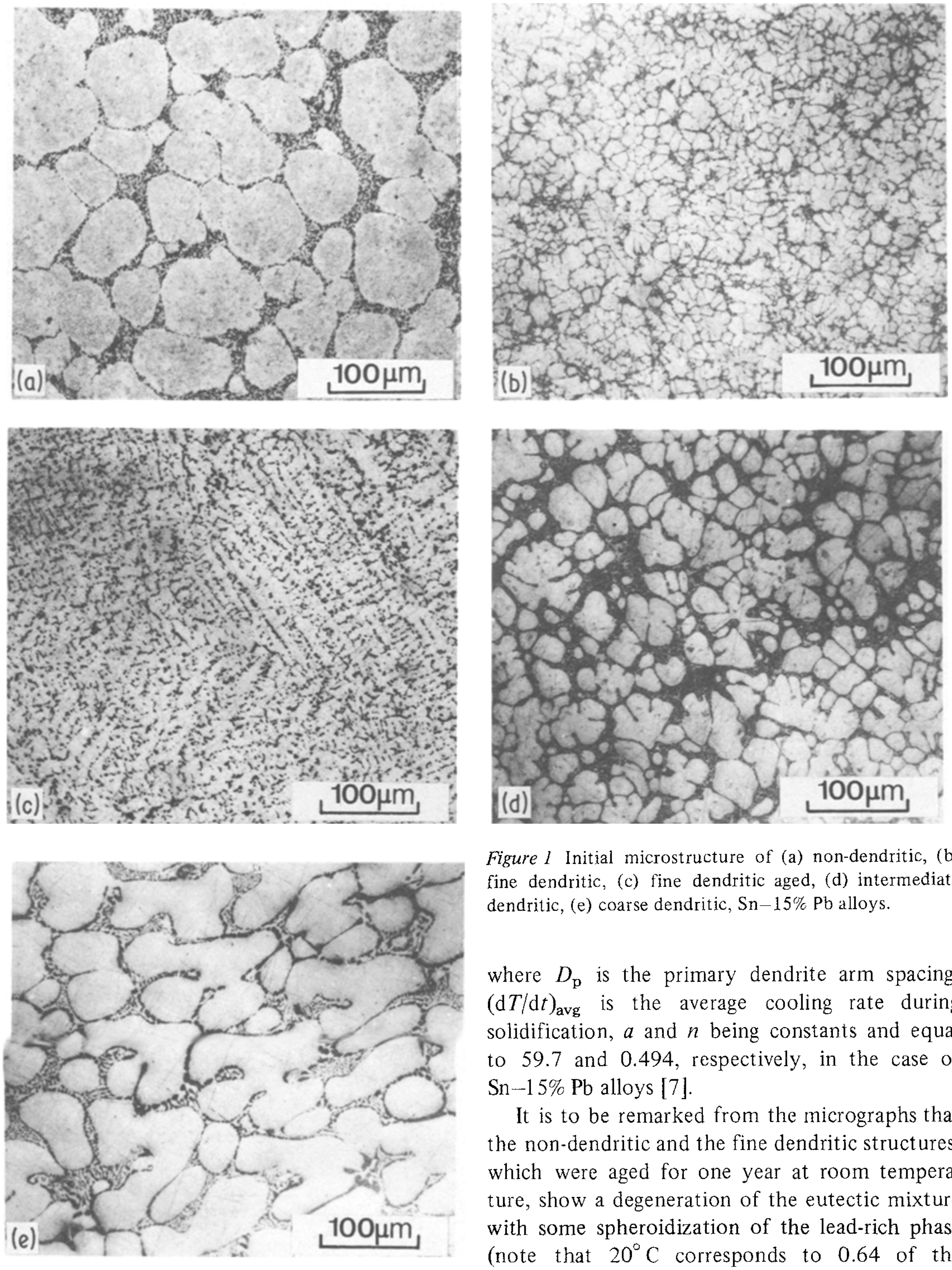


Figure 1 Initial microstructure of (a) non-dendritic, (b) fine dendritic, (c) fine dendritic aged, (d) intermediate dendritic, (e) coarse dendritic, Sn–15% Pb alloys.

The dendritic structure shows more or less equiaxed dendrites of the primary phase whose size was found to depend on the solidification rate according to the well-known relation

$$D_p = a[(\mathrm{d}T/\mathrm{d}t)_{avg}]^{-n} \qquad (1)$$

where D_p is the primary dendrite arm spacing, $(\mathrm{d}T/\mathrm{d}t)_{avg}$ is the average cooling rate during solidification, a and n being constants and equal to 59.7 and 0.494, respectively, in the case of Sn–15% Pb alloys [7].

It is to be remarked from the micrographs that the non-dendritic and the fine dendritic structures, which were aged for one year at room temperature, show a degeneration of the eutectic mixture with some spheroidization of the lead-rich phase (note that 20° C corresponds to 0.64 of the absolute melting temperature of the eutectic).

Fig. 2 is a plot of stress against strain for the non-dendritic and dendritic Sn–15% Pb specimens deformed at an initial strain-rate of 3.33×10^{-2} $\sec^{-1}$ at 184° C after a holding time of 150 sec before compression. Stress is defined here as the

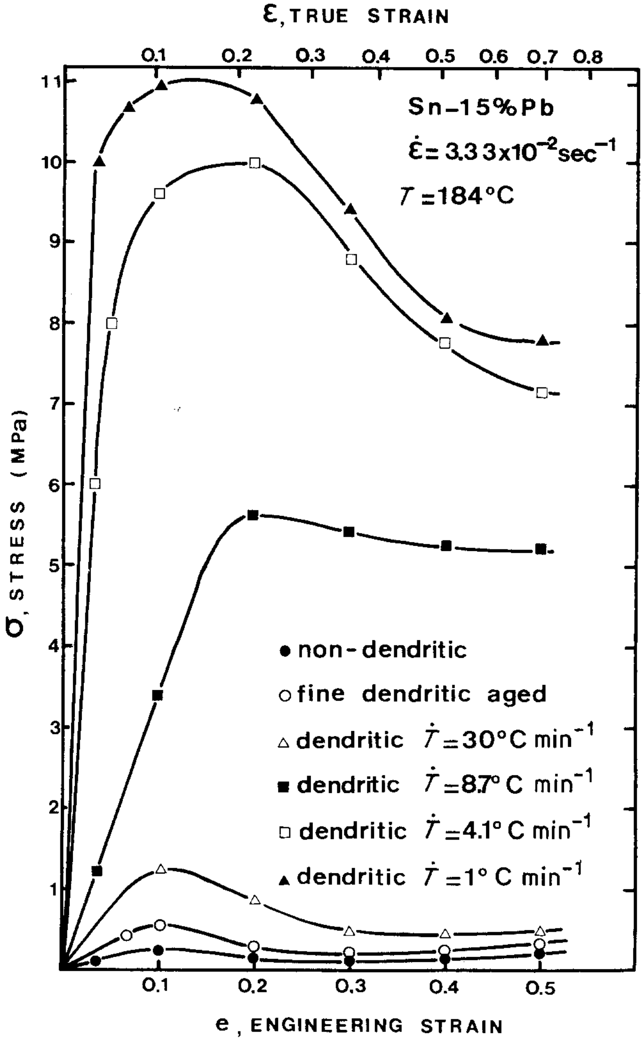


Figure 2 Stress against strain for coarse, intermediate, fine and non-dendritic Sn–15% Pb specimens deformed at an initial strain-rate of $3.33 \times 10^{-2}\,\mathrm{sec}^{-1}$ at 184° C. $\dot{T}$ refers to the average cooling rate of the alloys.

average pressure applied on the specimen:

$$\sigma_{\mathrm{avg}} = F_{\mathrm{exp}} h_0(1-e)/V \qquad (2)$$

where h_0 is the initial height of the specimen, F_{exp} is the measured force given by the load cell of the testing machine, V is the volume of the specimen and e is the engineering strain.

The curves are very different according to the solidification conditions. The non-dendritic structure deforms under very low stresses which remain approximately constant up to 50% engineering strain. The fine dendritic sample behaves closely like the non-dendritic one, with, however, a small maximum of stress at 0.1 strain. For the intermediate and coarse structures the stress is much higher; it increases as the cooling rate decreases, and for the two coarsest structures a maximum is seen in the curve at about 0.1 to 0.2 strain.

In opposition to what occurs in the semi-solid state, the compressive behaviour of the non-dendritic and the dendritic structures is identical in the solid state. Indeed, whatever the temperature is (20, 100 or 175° C), the stress–strain curves are almost the same for the different samples and this result can be explained by the relative independence of the structure size on the constitutive law in the hot deformation region. The stress level normally decreases as the deformation temperature increases. This is shown in Fig. 3 which is a plot of stress (taken at 0.3 engineering strain) against temperature for specimens deformed at an initial strain-rate of $3.33 \times 10^{-2}\,\mathrm{sec}^{-1}$. In the figure are also plotted for comparison the values of stress at 184° C for the structures under study. It is then clearly shown that, for the coarsest dendritic structures, the stress level

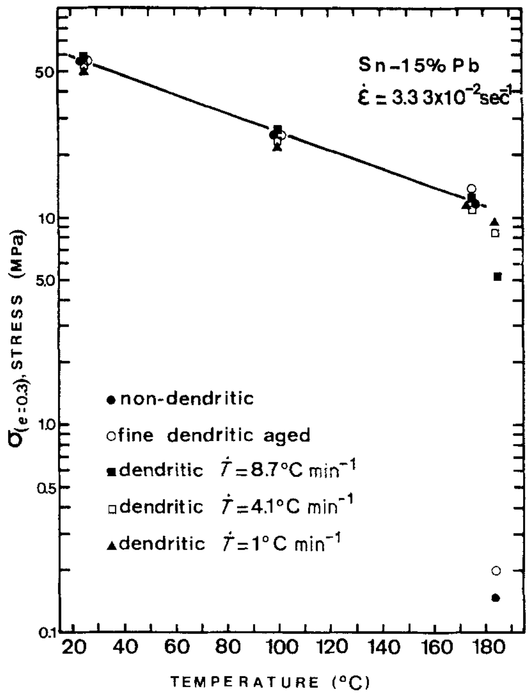


Figure 3 Stress (taken at 0.3 engineering strain) against temperature for Sn–15% Pb specimens deformed at an initial strain-rate of $3.33 \times 10^{-2}\ sec^{-1}$.

in the semi-solid state is about the same as that in the solid state, whereas for the fine and the non-dendritic structures, the stress level for the semi-solid state is more than 70 times smaller than that in the solid state.

During compression of semi-solid samples, segregation of the eutectic liquid occurs. This segregation was already reported and quantified in a non-dendritic and a coarse dendritic structure [5]. Fig. 4 shows the fraction of eutectic (liquid) g_L, measured along the radius of specimens of the alloys under study deformed at $3.33 \times 10^{-2}\ sec^{-1}$ at different strains. On the figure is also plotted the theoretical radius R assuming conservation of the whole volume (solid and liquid) of the specimen.

From the plots of Fig. 4, it can be concluded that:

(a) the segregation increases with the amount of compression;

(b) the segregation is more important at a given strain for coarse dendritic structures than for fine and intermediate ones;

(c) non-dendritic and fine dendritic structures deform almost homogeneously up to a compression of 70%;

(d) for the coarsest structures, the fraction liquid becomes zero in the central part of the specimen after a compression of 70%.

It is to be noted that no significant change in the amount of eutectic was found across the height of the samples and all the experimental points reported in Fig. 4 are the results of several measurements performed over this height.

All the previous results were obtained with specimens heated for 150 sec before compression. The effect of the holding time at 184° C before compression was investigated on the different structures, and Fig. 5 shows plots of stress (taken at 0.3 engineering strain) against holding time for Sn–15% Pb specimens deformed at an initial strain-rate of $3.33 \times 10^{-2}\ sec^{-1}$. The holding time, t_h, is defined here as the total time before compression including the heating time of the sample (90 sec) and the isothermal waiting time at 184° C. It is to be noted that the compression time for 30% engineering strain is only 9 sec, which is negligible compared to the holding time.

For the initially non-dendritic structure, stress decreases continually with holding time up to about 10 min, then it stabilizes at a very low level of 0.03 MPa. Fig. 6 shows optical micrographs of undeformed specimens held for 10, 30 and 180 min at 184° C, then quenched. It is clearly shown that the primary particles have coarsened owing to the time spent at high temperature with, in addition, some individualization and spheroidization.

For the intermediate dendritic structure, stress at 0.3 engineering strain also decreases with increasing holding time and it reaches almost the same stress level as for the non-dendritic structure after about 300 min. Fig. 7, which represents the structure of the alloy after 180 min holding time at 184° C, shows that the stress decrease is associated with a complete transformation of the dendritic into a non-dendritic type structure; this transformed structure closely resembles that for the initially non-dendritic alloy after a holding time of 180 min (see Fig. 6c).

For the coarse dendritic structure, holding time has only a very small influence on the stress level, this influence being even smaller for the coarsest one solidified at $1°C\ min^{-1}$.

For the initially fine dendritic structure, which was, however, aged for one year at room temperature before partial remelting, the stress evolution is the same as that for the non-dendritic up to a holding time of 4 min; stress increases

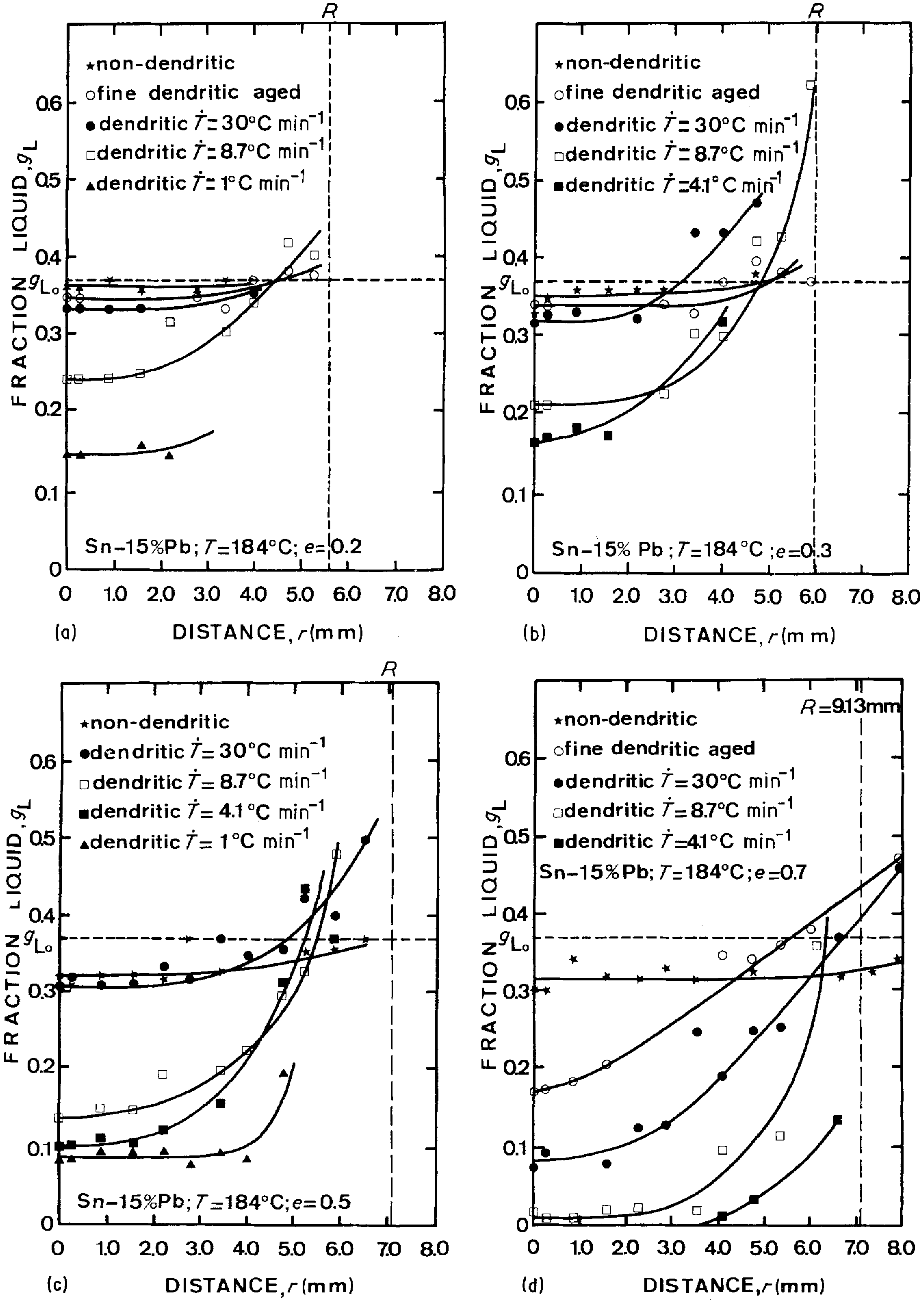


Figure 4 Fraction of liquid against distance along the radius for specimens of coarse, intermediate, fine and non-dendritic Sn–15% Pb deformed by compression at an initial strain-rate of 3.33×10^{-2} sec^{-1} up to different engineering strains: (a) $e = 0.2$, (b) $e = 0.3$, (c) $e = 0.5$, (d) $e = 0.7$.

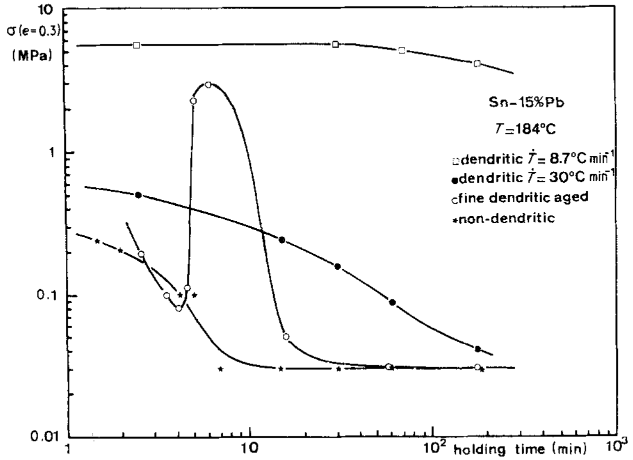


Figure 5 Stress (taken at 0.3 engineering strain) against holding time for non-dendritic coarse, intermediate and initially fine dendritic Sn–15% Pb specimens deformed at an initial strain-rate of 3.33×10^{-2} sec^{-1} after isothermal holding at 184° C.

thereafter very sharply up to a stress close to that for the coarse dendritic structure and decreases again to a value of 0.03 MPa after 30 min. Microstructural observations of undeformed specimens show that this stress variation is associated with: firstly, spheroidization of the fine dendritic structure (Fig. 8a), marked coalescence of the solid phase with large volume fraction of entrapped liquid leading to the formation of big, irregularly shaped particles (Figs. 8b and c), division of these big particles into smaller ones (Fig. 8d) and finally complete spheroidization of the structure (Fig. 8e).

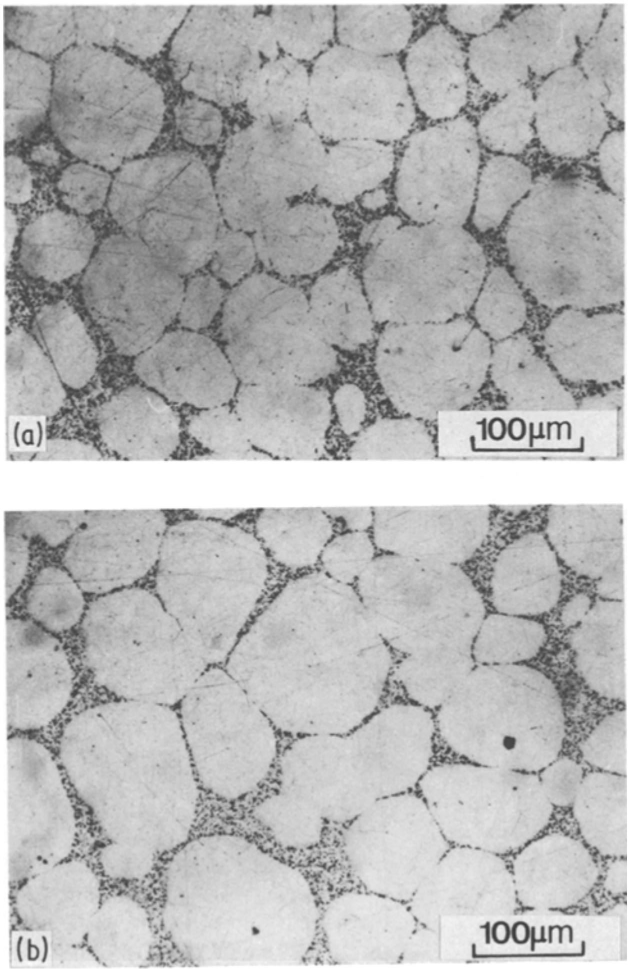


Figure 6 Microstructures of undeformed non-dendritic Sn–15% Pb specimens held at 184° C for different times: (a) $t_h = 10$ mn, (b) $t_h = 30$ mn, (c) $t_h = 180$ mn.

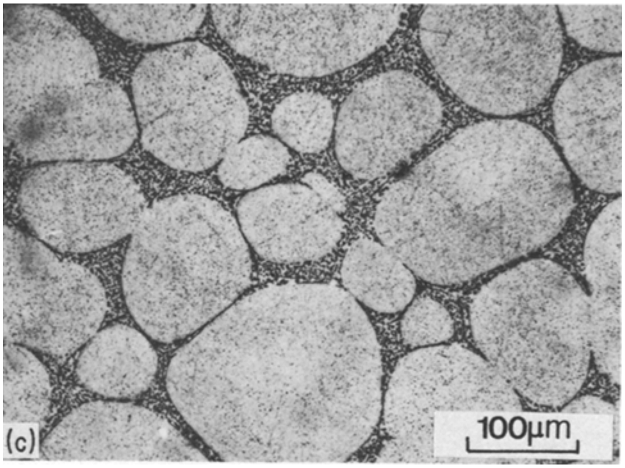


Figure 6 Continued.

Note that, as for the intermediate dendritic structure, some liquid still remains entrapped in the spherical tin-rich particles.

4. Discussion

4.1. Influence of the initial structure on the compression stress and deformation mode

The stress level during compression at constant velocity of semi-solid Sn–Pb specimens is strongly influenced by the structure of the solid phase, and two main types of behaviour can be observed, depending on the shape and the size of the structure.

For intermediate and coarse dendritic structures, the stress level is relatively high and it increases as the solidification rate decreases; this increase can be explained by the stronger interlocking of the dendrites as the structure becomes coarser, and in this case deformation takes place with some breakdown of the structure, which leads to an important stress decrease after 0.1 to 0.2 compression, as is clearly observed for the two coarsest structures. The maximum of stress is furthermore shifted to smaller strains for coarser structures. During deformation of the dendrites, liquid flow out of the sample occurs, leading to a high degree of segregation of the eutectic at the outer part of the compressed specimen. For the intermediate dendritic structure, interlocking of the dendrites is less, easier deformation can take place with some possible independent movements of the dendrites, one with respect to another [8], and this explains the smaller segregation of the eutectic liquid, and the small peak in the stress level observed after 10 or 20% compression.

For the fine and non-dendritic structures, the stress level is very low and almost no segregation of the liquid occurs, at least at low strains.

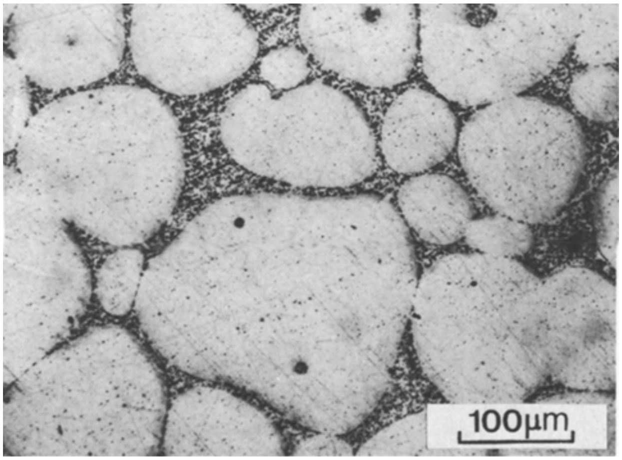
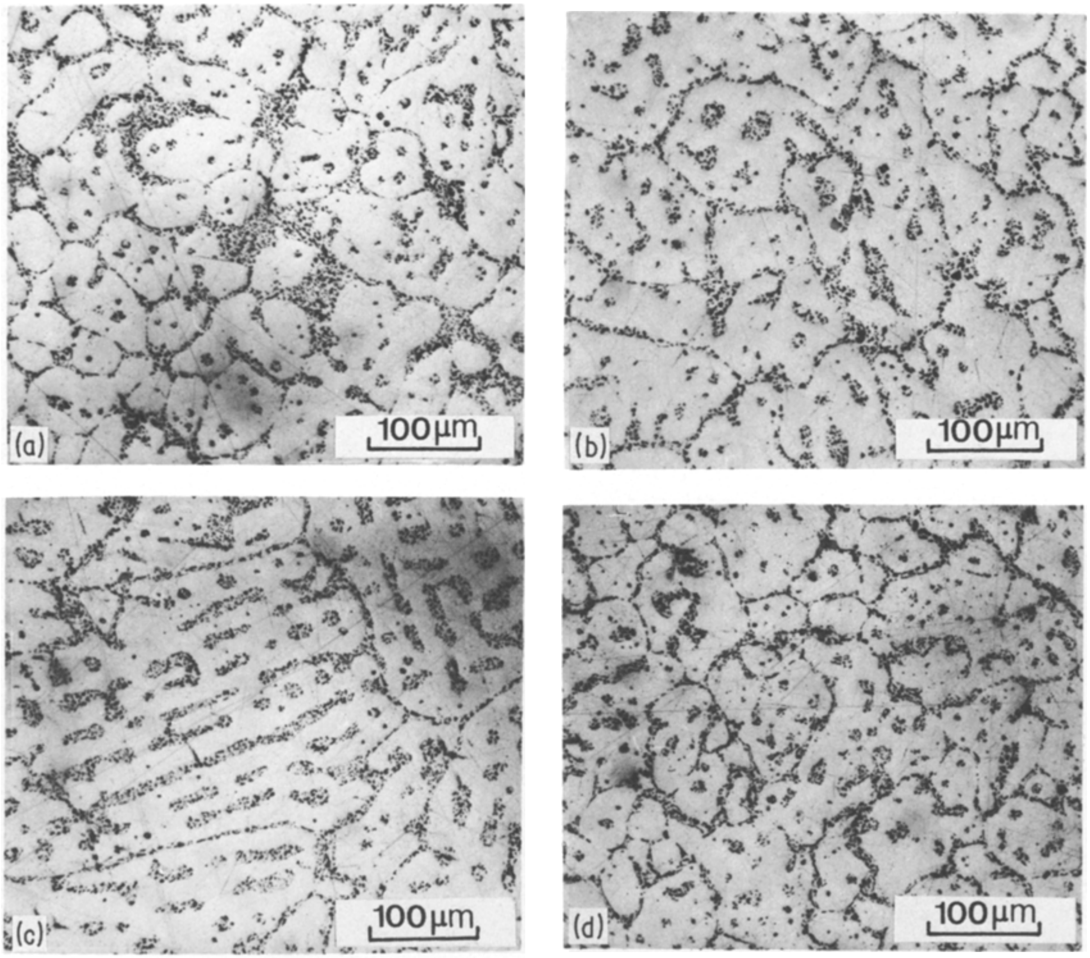


Figure 7 Microstructure of initially intermediate dendritic Sn–15% Pb specimen, held for 3 h at 184° C.



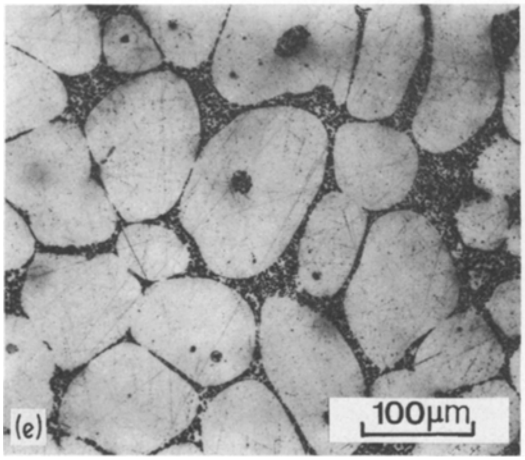


Figure 8 Microstructures of initially fine dendritic Sn–15% Pb specimens held at 184° C for different times: (a) $t_h = 3.5$ mn, (b) $t_h = 4.5$ mn, (c) $t_h = 6$ mn, (d) $t_h = 15$ mn, (e) $t_h = 30$ mn.

This absence of segregation can be explained by the shape of the primary particles which are almost spherical and remain essentially isolated from each other (except at very large strains) so that they can easily slide past one another without appreciable deformation. It is necessary to remember here that the curves of Fig. 2 were obtained after a heating time of 150 sec before compression, this time being sufficient to induce the first spheroidization of the fine dendritic structure.

4.2. Influence of the holding time before compression

For the initially non-dendritic structures, the decrease in the stress level with holding time before compression is associated with prominent individualization and spheroidization of the primary particles which are accomplished after about 10 min at 184° C. Coarsening of these particles also occurs, but this phenomenon is

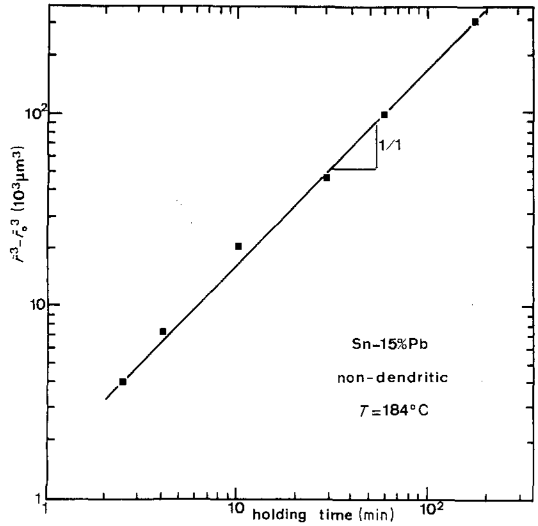


Figure 9 Change in the instantaneous average radius of the primary solid particles of non-dendritic Sn–15% Pb alloy during isothermal holding at 184° C.

found to have no influence on the stress level, which remains constant at 0.03 MPa for larger holding times. This constancy of stress can be explained by the fact that the size of the particles remains small compared to the size of the compressed specimen.

The evolution of the particle size with holding time at 184° C was determined, and Fig. 9 shows a plot of $r^3 - r_0^3$ against holding time t_h on a log–log scale, where r and r_0 are the instantaneous and initial average radius of the primary particles, respectively. The straight line of slope 1 indicates that coarsening occurs according to the Lifshitz, Slyozov and Wagner relation [9, 10]

$$r^3 - r_0^3 = kt \qquad (3)$$

where k is a constant equal to $2.75 \times 10^{-11}\,\text{cm}^3\,\text{sec}^{-1}$ in the case of Sn–15% Pb slurries. This law was found also to apply for describing coarsening of tungsten grains in a liquid nickel–tungsten matrix [11] and cobalt grains in a liquid copper matrix [12].

For the dendritic alloys, the effect of the holding time is different according to the initial size of the structure. It can, however, be understood at least during the first stage of the reheating process, by considering the critical time t_c for the evolution of a dendritic structure when maintained in the semi-solid state. This process was studied by Kattamis *et al.* [13] and is explained in terms of dissolution of small dendrite arms which form initially and later become unstable during solidification, surface energy reduction being the driving force for the process. It was found that t_c is given by the relation

$$t_c = \rho_s H C_L(1-k) m_L d^3/\sigma D_L T_L \qquad (4)$$

where ρ_s is the solid density, H the heat of fusion, m_L the slope of the liquidus, C_L the weight fraction of solute in the liquid at the equilibrium, k the partition ratio, d the dendrite arm spacing, σ the liquid–solid surface energy, D_L the diffusion coefficient in the liquid and T_L the equilibrium liquidus temperature.

Estimation of t_c for Sn–15% Pb shows that t_c is of the order of 2 to 10 min for the finest structure, 2 to 10 h for the intermediate structure and several days for the coarse dendritic structures. These results explain the rapid evolution of the fine dendritic structure and the corresponding decrease of the stress level during the first stage of the reheating process, and also the much slower evolution for the intermediate dendritic structure. In this case the stress level decreases slowly with increasing holding time and approaches that for the non-dendritic structure after about 3 h. For the coarse dendritic structure, however, there is almost no evolution of the dendrites for holding times smaller than 3 h and this explains the relative constancy of the stress level with holding time.

The first evolution of the fine dendritic structure characterized by coarsening and spheroidization of the dendrites leads to a very unstable microstructure; in the second stage of the annealing in the semi-solid state, marked coalescence of the solid phase occurs with a large amount of entrapped liquid leading to a drastic increase of the stress level. Indeed, very large, irregularly shaped particles can less easily move, one with respect to another, than can fine and spherical particles, this movement being even more difficult because of the reduction of the volume fraction of the interparticle (effective) liquid. The reduction of the interparticle liquid is clearly seen in Fig. 10 which shows that it is only one-half of the initial value after about 6 min holding time. The reason for this coalescence of the particles is not obvious. One explanation can, however, be put forward dealing with the progressive return of the structure to equilibrium. Indeed, during solidification at relatively high cooling rate, no solid diffusion is assumed to take place so that the fraction of the eutectic liquid is given by the

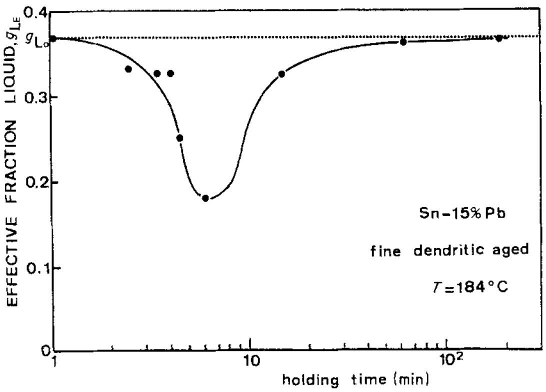


Figure 10 Evolution of the effective liquid fraction with holding time at 184° C for initially fine dendritic Sn–15% Pb specimens.

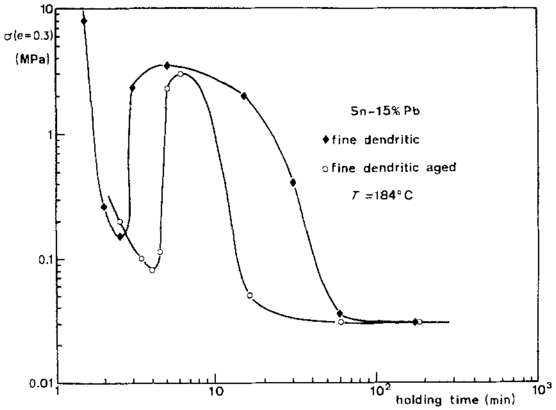


Figure 11 Stress (taken at 0.3 engineering strain) against holding time for as-cast fine dendritic and aged fine dendritic Sn–15% Pb specimens deformed at an initial strain-rate of 3.33×10^{-2} sec^{-1} after holding at 184° C.

Scheil equation

$$g_{Es} = (C_E/C_0)^{1/(k-1)} \tag{5}$$

where C_E and C_0 are the eutectic and initial compositions, respectively; calculation of g_{Es} gives a value of 0.37. This amount is also the fraction of liquid generated during partial remelting of the alloy. For holding times large enough to allow diffusion in the solid phase to take place, the fraction of liquid becomes, according to the lever rule,

$$g_{Ee} = (C_0 - C_{SM})/(C_E - C_{SM}) \tag{6}$$

where C_{SM} is the maximum solubility of lead in solid tin at the eutectic temperature. Calculation of g_{Ee} gives 0.35, which means that the return to equilibrium involves the separation of 2% eutectic liquid. This very small amount may perhaps be sufficient to explain the coalescence of the particles and the formation of large aggregates containing entrapped liquid, especially if it is in the form of narrow films. These narrow films of liquid might, moreover, be very unstable and tend to spheroidize, leading to additional agglomeration of the initially formed particles.

After the agglomeration stage, which corresponds to an important increase of the stress level during compression, a secondary spheroidization takes place and the stress decreases very sharply to a value of 0.03 MPa (i.e. by a factor of about 100) and stabilizes thereafter. This stress level seems then to be the intrinsic response of a completely spheroidized semi-solid Sn–15% Pb specimen with 0.65 solid volume fraction compressed at an initial strain-rate of 3.33×10^{-2} sec^{-1}. The driving force for this final spheroidization is probably the reduction of the liquid–solid boundary energy. Coarsening occurs also for this structure, but, as for the initially non-dendritic material, it is found to have no influence on the stress level during compression.

Results presented so far for the fine dendritic alloy were obtained with specimens which were aged for one year at room temperature before partial remelting and compression. The structural evolution which has taken place during this time was clearly seen in Fig. 1 which compares the as-cast structure with that obtained after ageing. For the latter, there is a marked degeneration of the eutectic mixture and the structure consists of lead particles finely dispersed in the tin matrix.

Fig. 11 shows plots of stress (taken at 0.3 engineering strain) against holding time at 184° C for fine dendritic specimens deformed at an initial strain-rate of 3.33×10^{-2} sec^{-1}. The curves are of the same general aspect whatever thermomechanical treatment is applied to the initial structure. The kinetics is, however, different: a delay is observed for the first spheroidization in the case of the aged structure, which can be explained by the fact that this ageing has led to a structure closer to equilibrium. The final spheroidization occurs earlier for the aged structure than for the as-cast structure but the reason for this is not known. It is to be noted that the stresses are almost identical for a holding time of 150 sec, which would probably indicate a similarity of structure and then of segregation behaviour for this holding time.

The possible evolution of fine dendritic structures into spheroidized ones, characterized by very low compression stresses in the semi-solid state equivalent to those observed for initially non-dendritic material, seems to be of great technological importance. Indeed, forming processes such as forging and extrusion can take advantage of these very low stresses, which are more than 100 times smaller than that observed in the fully solid state at about the same temperature. For these structures, however, the holding time in the semi-solid state before deformation has to be carefully selected in order to develop the first spheroidized structure which realizes a good optimization of the treatment in terms of time and strength of the developed structure.

The treatment can also be carried out in order to achieve the final spheroidization of the structure and in this case the minimum time necessary for this spheroidization to be completed is the important parameter.

5. Conclusions

The study in compression of Sn–15% Pb was conducted with specimens of coarse, intermediate, fine and non-dendritic structures. Results show that:

1. In the solid state, the compressive behaviour is independent of the solidification conditions.

2. In the semi-solid state, the behaviour depends closely on the structure of the solid phase and its evolution during the holding time of the sample before compression:

(a) Coarse structures with strong interlocking of the solid phase deform under high stresses and show an important segregation of the liquid phase during compression. Increasing holding time before deformation has almost no influence on the stress level.

(b) Initially non-dendritic or spheroidized dendritic structures deform under very low stresses without appreciable segregation of the liquid phase. In the completely spheroidized condition, the size of the particles has no influence on the stress level.

3. Observations concerning stress level and liquid segregation are technologically important for the industrial development of metal forming processes in the semi-solid state such as forging and extrusion. Rheocast as well as transformed dendritic materials can be used advantageously in such processes.

4. Stress during compression appears to be a very sensitive parameter of the morphology and the shape of the solid phase in partially remelted materials since stress variations of the order of 100 can be observed with alloys having the same initial microstructure but after different thermomechanical treatment. This factor can be even larger for alloys which have exactly the same composition but which have been solidified under different conditions.

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References

1. P. A. JOLY and R. MEHRABIAN, *J. Mater. Sci.* **11** (1976) 1393.
2. M. C. FLEMINGS, Rheocasting, MCIC 78-35 (1978).
3. C. MILLIÈRE, J. F. SECONDÉ, P. SÉCORDEL and M. SUÉRY, *Mém. et Etudes Sci. Rev. Mét.* June, July–August **80** (1983) 313, 355.
4. V. LAXMANAN and M. C. FLEMINGS, *Metall. Trans.* **11A** (1980) 1927.
5. M. SUERY and M. C. FLEMINGS, *ibid.* **13A** (1982) 1809.
6. M. C. FLEMINGS, Machine Casting of Ferrous Alloys, AMMRC CTR 75-22 (1975).
7. S. D. RIDDER, F. C. REYES, S. CHAKRAVORTY, R. MEHRABIAN, J. D. NAUMAN, J. H. CHEN and H. J. KLEIN, *Metall. Trans.* **9B** (1978) 415.
8. S. A. METZ and M. C. FLEMMINGS, *Trans. AFS* 77 (1969) 329.
9. I. M. LIFSHITZ and V. V. SLYOZOV, *J. Phys. Chem. Solids* **19** (1961) 35.
10. C. WAGNER, *Z. Elektrochem.* **65** (1961) 581.
11. T. K. KANG and D. N. YOON, *Metall. Trans.* **9A** (1978) 433.
12. C. H. KANG and D. N. YOON, *ibid.* **12A** (1981) 65.
13. T. Z. KATTAMIS, J. C. COUGHLIN and M. C. FLEMINGS, *Trans. Met. Soc. AIME* **239** (1967) 1504.

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