

Relationship between loss-modulus and homologous temperature in superplastic alloys

E. Elizabeth Martínez-Flores · Jesús Negrete · Gabriel Torres-Villaseñor

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Abstract A study of loss modulus values was conducted for three different metal alloys, in both superplastic and non-superplastic condition, using Dynamic Mechanical Analysis (DMA). Results showed a direct relationship between loss modulus values and the homologous superplastic temperature for each of the three different metal alloys that were studied.

Keywords DMA · Homologous temperature · Superplasticity

Dynamic Mechanical Analysis was conducted with TA Q800 DMA equipment, using a single cantilever clamp. The three metal alloys used were Zn-21wt.% Al-2wt.% Cu (Zn-21Al-2Cu), Cd-17wt.% Zn (Cd-17Zn) and Sn-38wt.% Pb (Sn-38Pb). Specimens used were non-superplastic (as-cast) plates and superplastic (rolled sheet) plates, measuring $4 \times 1 \times 35$ mm. Tests were carried out with an oscillation amplitude of 15 μm , with temperature sweeps

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E. E. Martínez-Flores (✉)
Facultad de Ingeniería, UASLP, Dr. Manuel Nava No. 8,
Zona Universitaria, 78290 San Luis Potosí, S. L. P., México
e-mail: emartine@uaslp.mx

J. Negrete
Instituto de Metalurgia, UASLP, Sierra Leona 550, Lomas 2a.
Sección, 78210 San Luis Potosí, S. L. P., México

G. Torres-Villaseñor
Instituto de Investigaciones en Materiales, UNAM,
Apdo. P 70-360, 04519 México, D. F., México

between 25–300 °C for Zn-21Al-2Cu, 25–200 °C for Cd-17Zn and 25–140 °C for Sn-38Pb. All tests were conducted at a constant frequency of 1.0 Hz, with heating rates of 3 °C/min. Findings were analyzed using TA Universal Analysis 2000 software.

The Zn-22wt.% Al and Zn-21wt.% Al-2wt.% Cu alloys behaved superplastically in the range of 473–533 K (0.63–0.70Tm) [1–5]. Pb-62wt.% Sn reached an exceptional superplasticity level at 413 K (0.90Tm) [5–7]. For Cd-17wt.% Zn, an extensive grain boundary activity was reached at 377 K (0.69Tm) [8].

Dynamic Mechanical Analysis (DMA) has been widely used in polymer characterization [9–15]. Recently, DMA has begun to be used as an alternative technique for measuring viscoelastic and damping properties of metal alloys [16–21]. During testing, a metal plate is subjected to a sinusoidal stress (σ). The resulting strain (ε) is also sinusoidal with the same frequency, but with a lag in the phase. This phase lag is expressed as an angle, δ ; the tangent of the phase angle δ is a measure of the energy dissipation or damping of the material. The modulus is the ratio between the stress and the strain. For a viscoelastic material, the modulus is a complex quantity, E^* [10, 22]

$$E^*(\omega) = E'(\omega) + iE''(\omega)$$

E' represents the storage modulus, while E'' represents the loss modulus. The storage modulus, E' , reflects the elastic response of the material. Loss modulus, E'' , describes the strain energy that is completely dissipated or lost as a result of friction and internal motions. Such changes indicate the occurrence of internal friction produced by microstructural grain mobility, and the capability of microstructural domain reorientation of the material. In the case of polycrystalline metal, neighboring grains may have different amounts of misorientation when the specimen is deformed.

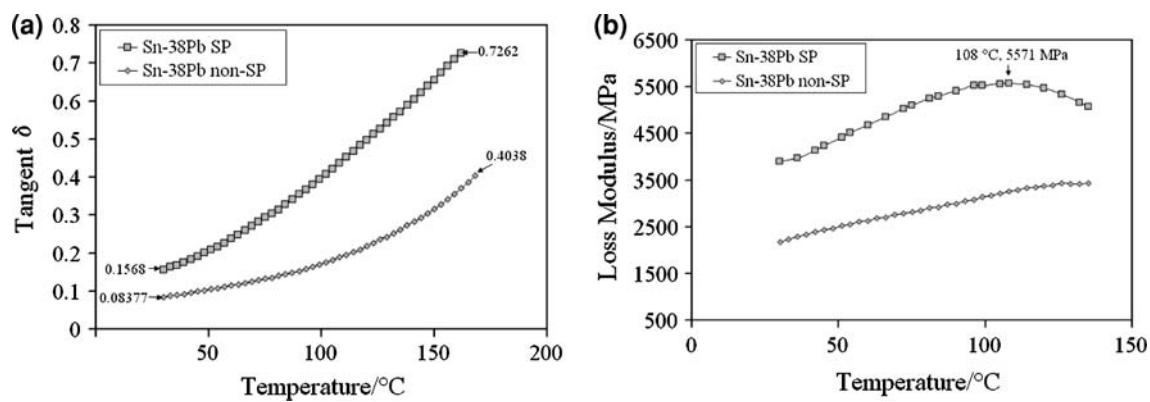


Fig. 1 Results of dynamic mechanical analysis carried out in both superplastic and non-superplastic Sn-38wt.% Pb alloy. Curve (a) shows $\tan \delta$ and curve (b) Loss Modulus, both as function of temperature

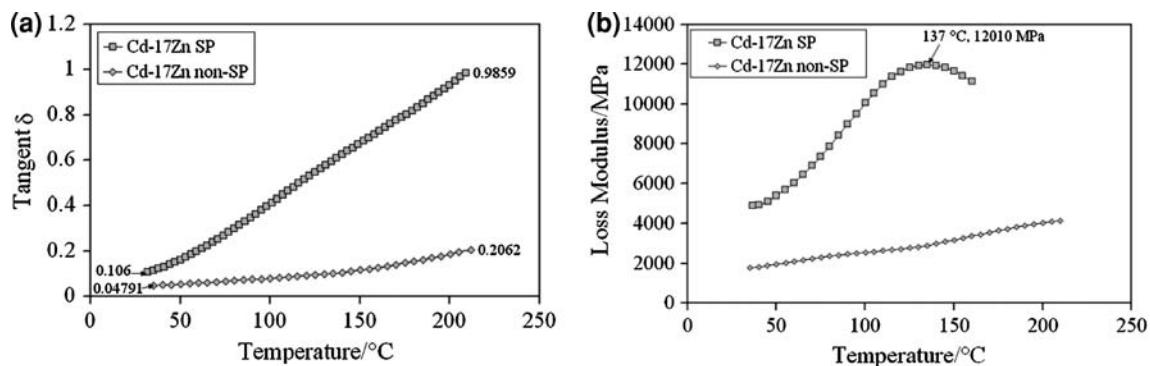


Fig. 2 Results of dynamic mechanical analysis carried out in both superplastic and non-superplastic Cd-17wt.% Zn alloy. Curve (a) shows $\tan \delta$ and (b) Loss Modulus, both as function of temperature

The capability of internal displacements between grain boundaries are related to the superplastic behavior of the material.

Results of loss modulus and $\tan \delta$ as a function of temperature for alloys in this study, in both superplastic and non-superplastic condition, are presented in Figs. 1, 2 and 3. Curves in the Figs. 1a, 2a and 3a show results of $\tan \delta$ as a function of temperature. In all studied alloys, damping increase monotonically when temperature increases. These results were consistent with the behavior observed in samples tested in a torsion pendulum [23–28]. High values of $\tan \delta$ were observed in both superplastic and as-cast alloys.

Loss modulus curves 1(b), 2(b) and 3(b) show that all superplastic alloys had a maximum value, at 108 °C for Sn-38Pb alloy, 137 °C for Cd-17Zn and 250 °C for Zn-21Al-2Cu, respectively. For comparison purposes, test results for non-superplastic alloys are included in each figure. No loss modulus peak was observed for any of the non-superplastic alloys.

Figure 4 shows the loss modulus values as a function of homologous temperature (T/T_m), in this ratio, T is the test temperature and T_m is the melting temperature both in

absolute scale. For each alloy we can observe a maximum in the curve at 0.83 for Sn-38Pb alloy, 0.69 for Zn-21Al-2Cu alloy and 0.75 for Cd-17Zn alloy respectively. These values of the ratio T/T_m coincide with the homologous temperatures reported for superplastic behavior of these alloys [1–8].

It has been well established that grain boundary sliding strongly depends on the type and the boundary misorientation angle. Watanabe [29] observed the influence of the boundary misorientation angle on sliding. Sliding takes place more significantly with increasing the misorientation angle up to 40° and then becomes difficult showing little sliding around 55°, due to the presence of the coincidence orientation relationships. Thus sliding is difficult at low-angle boundaries and coincidence boundaries, while it is easy at high angle boundaries. The evolution of the grain boundaries during superplastic deformation, have been observed by others authors [30, 31]. The superplastic state will be reached when a maximum of grain boundaries are ready for slip with minimum stress (a maximum of high angle boundaries), thus it is possible that the increase of the loss modulus is related with the accommodation of grain boundaries, in the elastic region, from non-sliding state to

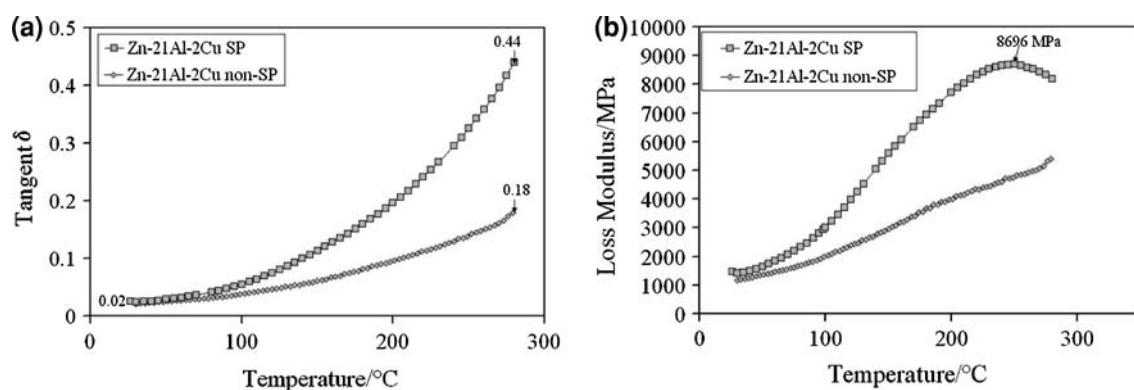


Fig. 3 Results of dynamic mechanical analysis carried out in both superplastic and non-superplastic Zn-21wt.% Al-2wt.% Cu alloy. Curve (a) shows $\tan \delta$ and (b) Loss Modulus, both as function of temperature

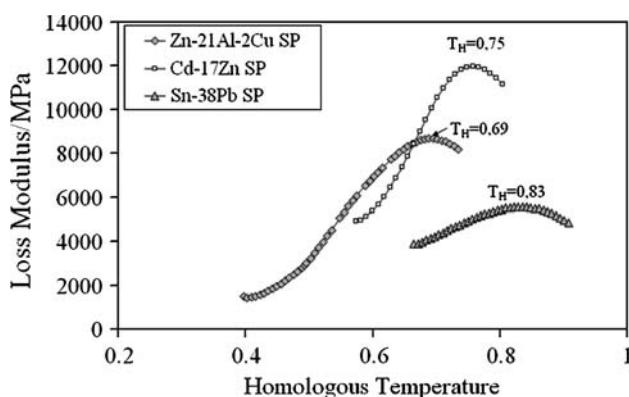


Fig. 4 Loss Modulus behavior as function of homologous temperature for the superplastic alloys

sliding state on plane where a maximum in shear stress exists. The required changes of the grain boundaries to achieve a superplastic state can be as follow: As result of thermal fluctuation, atoms at each grain boundary occasionally have sufficient energy for atomic diffusion at the grain boundaries in the absence of external forces. If an external force is applied however, the energy for diffusion can be lower in the direction of the maximum shear stress; consequently grains groups probably will shift as a whole along grain boundary planes oriented close to the maximum shear stress direction. The effect of the diffusion flow will be to dissipate part of the elastic energy stored in the solid, which on the macroscopic scale will appear as an increase of the loss modulus.

It is possible to observe from Figs. 1a, 2a, and 3a, that damping in superplastic alloys is considerably higher than in cast alloys, reaching values only observed in polymeric materials.

This study revealed the existence of a relationship between loss modulus maximum values and the temperature where a maximum superplastic deformation has been reported for Cd-Zn, Pb-Sn and Zn-Al superplastic alloys.

The results suggest the existence of a mechanism that accommodates the grain boundaries in the elastic region for easy slide in the plastic region.

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References

- Mohamed F, Mohamed M, Langdon T. Factors influencing ductility in the superplastic Zn-22 pct Al eutectoid. Metall Trans A. 1977;8A:933–8.
- Vale S, Eastgate D, Hazzledine P. The low strain rate behaviour of superplastic Zn-Al eutectoid alloy. Scr Metall. 1979;13:1157–62.
- Naziri H, Pearce R. The Influence of copper additions on the superplastic forming behaviour of the Zn-Al eutectoid. Int J Mech Sci. 1970;12:513–21.
- Stewart M. Superplastic forging of Zn-Al-Cu alloys. Can Metall Q. 1973;12(2):159–69.
- Mohamed F, Mohamed M, Langdon T. Exceptional ductility in the superplastic Pb-62 pct Sn eutectic. Metall Trans A. 1977;8A: 1832–4.
- Kashyapand B, Murty G. On the uniqueness of stress-strain rate relation for superplastic flow of the Pb-Sn eutectic. Metall Trans A. 1981;12A:1923–5.
- Valiev R, Langdon T. An investigation of the role of intragranular dislocation strain in the superplastic Pb-62% Sn eutectic alloy Acta Metall Mater. 1993;41(3):949–54.
- Merriman E. Superplastic deformation in a eutectic alloy of cadmium and zinc, Thesis, Illinois University, Urbana, 01 Jan 1970.
- Dawkins J, editor. Developments in polymer characterization—5. London and New York: Elsevier Applied Science Publishers; 1986. p. 179.
- Turi EA, editor. Thermal characterization of polymeric materials, vol. 1., 2nd ed. New York: Academic Press; 1997. p. 133.
- Menard KP. Dynamic mechanical analysis. A practical introduction. USA: CRC Press LLC; 1999. p. 61–9.
- Prolongo M, Arribas C, Salom C, Masegosa R. Mechanical properties and morphology of epoxy/poly(vinyl acetate)/poly(4-vinyl phenol)brominated system. J Therm Anal Calorim. 2007;87:1–33.

13. Szeluga U, Kurzeja L, Galina H. Dynamic mechanical properties of epoxy/novolac system modified with reactive liquid rubber and carbon filler. *J Therm Anal Calorim.* 2008;92(3):813–20.
14. Joshi R, Lefevre E, Patel C, Provder T, Crombez R, Shen W, et al. Thermoanalytical and morphological studies of cross-linked latex films by advanced techniques. *J Therm Anal Calorim.* 2008;93(1):19–26.
15. Mothé Ch, de Araujo CR, Wang S. Thermal and mechanical characteristics of polyurethane/curaua fiber composites. *J Therm Anal Calorim.* 2009;95(1):181–5.
16. Artiaga R, García A, García L, Varela A, Mier JL, Naya S, et al. DMTA study of a nickel–titanium wire. *J Therm Anal Calorim.* 2002;70:199–207.
17. Van Humbeeck J. Damping capacity of thermoelastic martensite in shape memory alloys. *J Alloy Compd.* 2003;355:58–64.
18. Bessegini S, Villa E, Portman J. DMA characterization of a Ni50.5at%Ti shape memory alloys. *Int J Appl Electromagn Mech.* 2006;23:33–8.
19. Gündüz S. An internal friction study of a vanadium microalloyed steel by a dynamic mechanical thermal analyser. *Turk J Eng Env Sci.* 2002;26:353–9.
20. Aaltio I, Lahelin M, Söderberg O, Heczko O, Löfgren B, Ge Y, et al. Temperature dependence of the damping properties of Ni-Mn-Ga alloys. *Mater Sci Eng A* (2007). doi:[10.1016/j.msea.2006.12.229](https://doi.org/10.1016/j.msea.2006.12.229).
21. Wang Q, Han F, Wu J, Hao G. Damping behavior of porous CuAlMn shape memory alloy. *Mater Lett.* 2007;61:2598–600.
22. Lakes RS. Viscoelastic solids. New York: CRC Press; 1998. p. 1–9; 63–109, 243–77.
23. Ritchie I, Pan Z, Goodwin F. Characterization of the damping properties of die-cast zinc-aluminium alloys. *Metall Trans.* 1991;22A:617–22.
24. Zelin MG, Krailinov NA, Valiev RZ, Grabski MW, Yang HS, Mukherjee AK. On the microstructural aspects of the nonhomogeneity of superplastic deformation at the level of grain groups. *Acta Metall Mater.* 1994;42:119–26.
25. Zhang P, Kong QP, Zhou H. Dynamic internal-friction study of the superplastic deformation in a Pb-Sn alloy. *Phil Mag A.* 1998;77(2):437–46.
26. Buechner P, Stone D, Lakes RS. Viscoelastic behavior of superplastic 37 wt% Pb 63 wt% Sn over a wide range of frequency and time. *Scripta Mater.* 1999;41(5):561–7.
27. Chuvil'deev VN, Nieh TG, Gryaznov MY, Kopylov VI, Sysoev AN. Superplasticity and internal friction in microcrystalline AZ91 and ZK60 magnesium alloys processed by equal-channel angular pressing. *J Alloy Compd.* 2004;378:253–7.
28. Arzhavitin VM. Amplitude dependence of the internal friction in a Pb-62% Sn alloy. *Tech Phys.* 2004;49(6):707–10.
29. Watanabe T. Key issues of grain boundary engineering for superplasticity. *Mater Sci Forum.* 1997;243–245:21–30.
30. McNelley TR, McMahon ME. An investigation by interactive electron backscatter pattern analysis of processing and superplasticity in an aluminum-magnesium alloy. *Metall Mater Trans.* 1996;27A:2252–62.
31. Perez-Prado MT, Cristina MC, Torralba M, Ruano OA, González-Doncel G. Texture gradient evolution in Al-5% Zn sheet alloy after tensile deformation at high superplastic strain rate. *Scr Mater.* 1996;35(12):1455–60.