

Determination of Recrystallization Temperature of Varying Degrees Formed Aluminium, by DMTA Technique

Zsolt Dugár, Péter Barkóczy, Gábor Béres, Dávid Kis, Attila Bata, Tamás Dugár, Zoltán Weltsch

Abstract—This study is about the structural transformations of aluminium examining with the Dynamic Mechanical Thermal Analyzer (DMTA). It is a faster and simpler measuring method to make consequence about the metal's structural transformations. The device measures the changing of the mechanical characteristics depending on the heating rate, and concludes certain transformations. This measuring method fast and shows clean-cut results comparing the conventional ways.

Applying polymer measuring devices for metal investigations is not widespread method. One of the adaptable ways is shown in this study. The article compares the results of the small specimen test and the DMTA method, considering the temperature and the forming dependence of recrystallization temperature.

Keywords—DMTA, recrystallization, cold forming, rotation.

I. INTRODUCTION

MANUFACTURING of aluminium strip is usually done by cold rolling. Due to the rolling, the material runs out of formability and the aluminium get hardened. For subsequent shaping annealing and recrystallization is necessary.

The temperature of the recrystallization highly depends on the grade of the subsequent shaping. If the recrystallization temperature is too high, or coupled with too long time, the particles might get rough. While at lower temperatures than required, the mechanical properties cannot become favourable, the hardness remains. Small specimen test is a current process to determine the appropriate temperature of recrystallization. The test requires many specimens, and lots of time. In this article it is presented a simpler and faster method to determine temperature of recrystallization [1].

For the accurate definition of the recrystallization temperature, the Dynamic Mechanical Thermal Analyser (DMTA) method - received from polymer science - gathers ground. The equipment measures the change of the mechanical characteristics, during given heating rate, and concludes certain transformations, based on the changes.

The transition peak typology finds its basis in the thermoelastic martensitic phase transformation due to the mobility of martensite variant interface and twin boundaries and is responsible of the damping properties of the material;

Zs. Dugar, Z. Weltsch, G. Béres, and D. Kis are with the Department of Materials Technology, Faculty of Mechanical Engineering and Automation, Kecskemét College, (address: H-6000 Kecskemét, Hungary, Izsáki út 10; e-mail: dugar.zsolt@gamf.kefo.hu, weltsch.zoltan@gamf.kefo.hu, beres.gabor@gamf.kefo.hu, kis.david@gamf.kefo.hu).

frequency does not alter the peak position but affects the peak height which is also influenced by the cooling rate. Moreover, if the material is held isothermal at the transformation peak temperature and the strain and the frequency are kept at a constant value, a reduction in IF peak as a function of time can be observed. The phase transition is also visible in the storage modulus curve; as a matter of fact, the maximum of the IF peak due to the phase transition corresponds to a minimum in the analogous storage modulus curve. [14].

There are many effects associated to a frequency variation in an anelastic material: thermo-elastic phenomena, movement of valence electrons, grain boundary viscosity, interstitial atoms movement, and dislocation relaxation. IF peak height and frequency position depends on the composition, on the sub structural state and on temperature. Probably due to the amount of features which respond to a frequency variation, a detailed frequency scanning of IF measurements at a constant temperature is scarcely employed in SMAs field and very few examples can be found in published literature [14].

The article compares the results of the small specimen test and the DMTA method, considering the temperature and the forming dependence of recrystallization temperature.

II. EXPERIMENTAL CONDITIONS

In order to see how the measurement method is applicable, it is prepared AlMg₃ specimens with different shaping grade. The rolling was done by duo roll stand, the shaping grade ranged from 30 to 90%. More differently shaped specimens were processed than the commercially available rolling states, to get more accurate measurements' results.

The initial thickness was 1mm in all cases. Before the last rolling, the specimens were annealed due to the homogenous structure, as suggested by the literature.

During cold rolling, the mechanical and physical properties of the metals change. The strength greatly increases, while ductility, and deformation capacity decreases. Of course, the properties depend on the shaping grade. This is because the crystal lattice distorted in the deformed particles and the dislocation density increased [2]-[4].

In the deformed crystal lattice, the atomic distance is changed from the original minimum energy state. Depending on the degree of displacement, the atoms get extra energy. Whereas, the distance among the atoms mostly determined by the shaping grade, it might say, that higher forming gets higher extra stored energy.

During the rolling, the grain boundaries, and the dislocations moves on, while new dislocations generated. The amount of dislocations is varied by the penetration, or the tearing out of atomic-lines. This symptom also involves the movement of the atoms, so the creation of the dislocations also causes an increase in energy. Furthermore, the increased number of dislocations prevents each other in the movement, following forming needs higher stress. This hardening is able to present by hardness testing (Fig. 1).

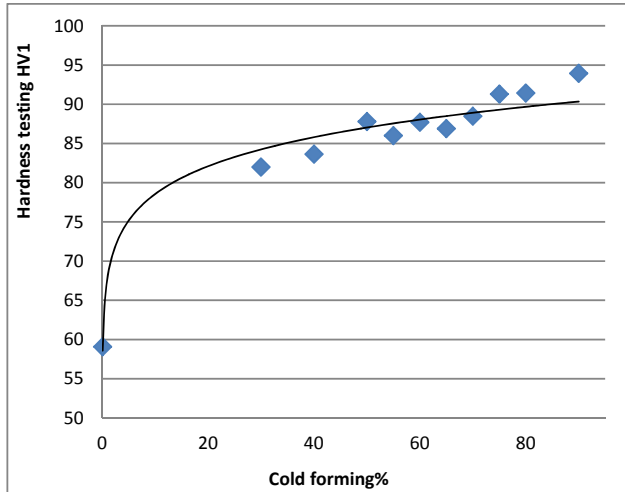


Fig. 1 Hardening of AlMg₃ due to rolling

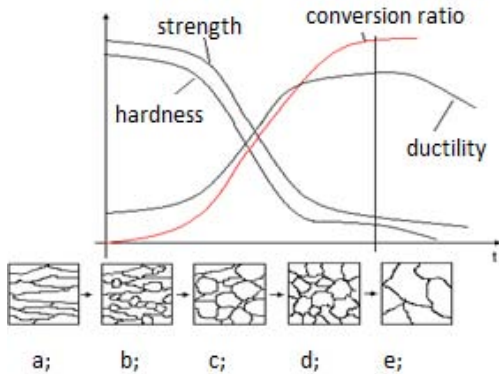


Fig. 2 Conformation of the granular structure and the mechanical properties during the recrystallization: The transformation ratio indicates the amount of recrystallized grains deformed grains, b) appearance of new seeds, c) growth of the grains, d) primer recrystallization, e) secondary recrystallization

The materials aim to reach the lowest-energy state. The raising of the temperature provides the opportunity to start the necessary diffusion process. The diffusion (thermally activated process) is stimulated by the energy state difference between the initial and the final conditions. Result of the annealing, the internal stress, the hardness, and the strength values return to the state before forming. The amount of dislocations also returns to its equilibrium value. In the structure of the material new seeds appear, and solid-state crystallization occurs. This

process is called recrystallization, shown by Fig. 2. Higher forming grade causes higher stored energy, which results the decrease of the transition temperature [3]-[9]. In this article, this temperature changing is examined.

DMTA was performed using TA Q800. This analyser provides 16 frequencies in the range from 0.01 Hz to 200 Hz at ten different strain levels corresponding to dynamic displacements between 5 μm and 128 μm . Fig. 3 illustrates experimental options provided by the instrument. All these deformation geometries are widely used for the analysis of polymers. For mechanical experiments on very stiff materials like network glasses, only the bending geometries are suitable, but neither the literature nor the DMTA handbook supply detailed experimental instructions for the measurement of mechanical losses due to ion transport. The best procedure, here, was arrived at by a process of trial and error.

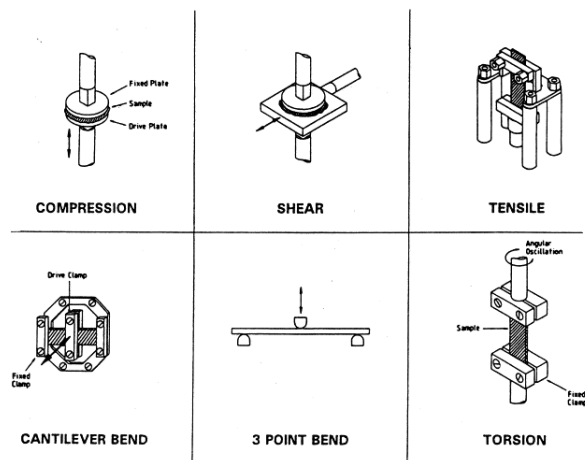


Fig. 3 The experimental configurations for DMTA [15]

The instrumentation of a DMA consists of a displacement sensor such as a linear variable differential transformer, which measures a change in voltage as a result of the instrument probe moving through a magnetic core, a temperature control system or furnace, a drive motor (a linear motor for probe loading which provides load for the applied force), a drive shaft support and guidance system to act as a guide for the force from the motor to the sample, and sample clamps in order to hold the sample being tested. Depending on what is being measured, samples will be prepared and handled differently.

Temperature sweep. A common test method involves measuring the complex modulus at low constant frequency while varying the sample temperature [17].

Frequency sweep. A sample can be held to a fixed temperature and can be tested at varying frequency. Peaks in $\tan\delta$ and in E'' with respect to frequency can be associated with the glass transition, which corresponds to the ability of chains to move past each other. Note that this implies that the glass transition is dependent on strain rate in addition to temperature. Secondary transitions may be observed as well.

Dynamic stress-strain studies. By gradually increasing the amplitude of oscillations, one can perform a dynamic stress-strain measurement. The variation of storage and loss moduli with increasing stress can be used for materials characterization, and to determine the upper bound of the material's linear stress-strain regime [16].

Small Specimen Test:

To determine the parameters of the recrystallization, experimental series were performed by hardness testing. The sampling temperature was grown by 50°C from 200°C to 300°C. Over 300°C, samples were taken in every 25°C (see Table I). Initially, the heating lasted for half an hour in every case, then the time was raised to one and half, and three hours, at the favourable temperature ranges.

The sample geometry was 50 x 50 mm, the forming grade was 80%. At other forming grades the results shown similar values, cause the inaccurate furnace temperature control.

The heat treatment was done by OH 63 chamber furnace, without protective atmosphere.

The small specimen tests revealed that annealing up to 250°C for 90 minutes did not cause relevant changes in hardness. However, stress relaxation might cause slight reduction of hardness values. Annealing on 300°C for 30 minutes reduces hardness significantly. Beyond this range the changes of annealing time and temperature do not affect to the specimen's hardness greatly.

The samples can be fixed in various clamps which can vibrate the specimen with a specified sinusoidal load and amplitude. The measurements were made in tensile and bending clamps. The clamps are placed in a furnace filled with neutral gas (nitrogen), which heats or cools, with a guided rate. Fig. 5 shows the DMTA results of a 90% formed sample.

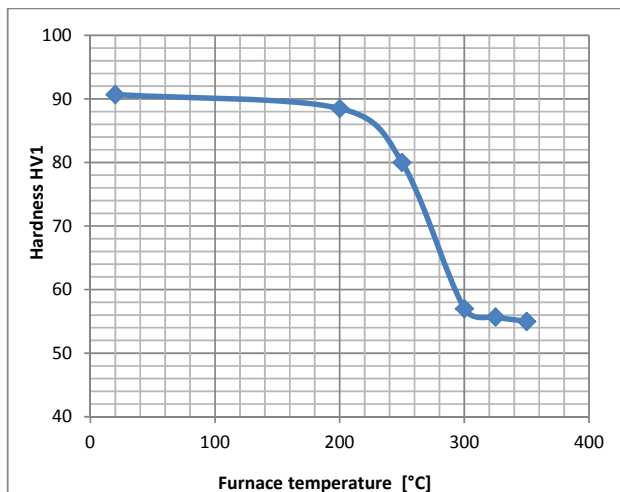


Fig. 4 Hardness test results in function of furnace temperature in case of AlMg₃ sample 30 minutes annealed

TABLE I
HARDNESS VALUES OF ALMG3 AFTER HEAT TREATMENT, AT 80% FORMING GRADE

Annealing temperature [°C]	Annealing time [min]	Measurement number HV ₁				Mean HV ₁
		1	2	3	4	
20	0	88.6	91.8	90.2	91.1	90
	30	87.7	88.8	88.5	88.9	88.5
200	60	85.1	85.3	84.1	86.7	85.3
	90	84.2	84	85.1	83.4	84.2
250	30	80.7	80.5	81	79.7	80.5
	60	81.4	79.4	78.5	79.8	79.8
300	90	79.2	79	78.9	79	79.0
	30	55.1	55.7	55.1	55.3	55.3
300	60	57.2	57.1	56.5	56.9	56.9
	90	57.5	55.5	56.3	56.4	56.4
325	30	54.9	55.7	56.4	55.8	55.7
	60	54.8	55.9	57.1	55.9	55.9
350	90	56.8	56.1	56.6	56.5	56.5
	30	54.5	54.7	55	54.7	54.7
350	60	57.3	57.3	57.2	57.4	57.3
	90	57.3	57.8	57	57.4	57.4

Fig. 4 shows the Vickers hardness test's results in function of isothermal temperatures, in case of 30 minutes annealing.

III. DMTA TEST RESULTS

The dynamic mechanical thermal analyzer (commonly known as DMTA) creates a direct link between the materials' chemical structure and mechanical properties. The transitions are generated by heating or cooling, among dynamic

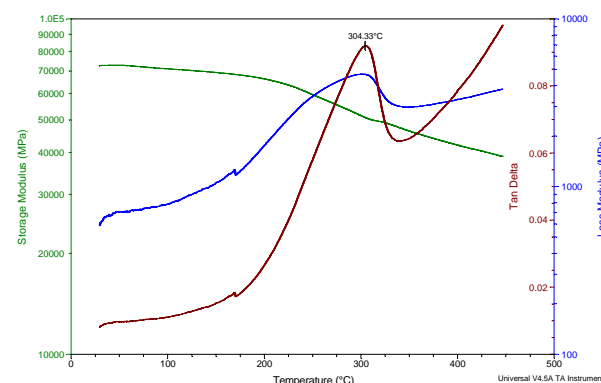


Fig. 5 DMTA curves of a 90% formed AlMg₃ sample

The dimensions of the sample are 18 x 5mm. The specimen was heated up to 450°C with 3°C/minute heating rate in a two point bending clamp. The frequency was set to 5 Hz, the deformation amplitude was 5 μm. The green curve shows the storage modulus of the material which is proportional to the Young modulus. The blue curve shows the loss modulus which originates from the internal friction during the transformation of the atomic structure. The quotient of these two values is the tanδ. The tanδ peak shows where the transformation occurs. In case of Fig. 5, the tanδ peak locates the recrystallization at 304.3°C.

Fig. 6 shows the discrepancy the tanδ curves of the 30% formed and 90% formed samples. The results show that the transformation temperature depends on the shaping grade.

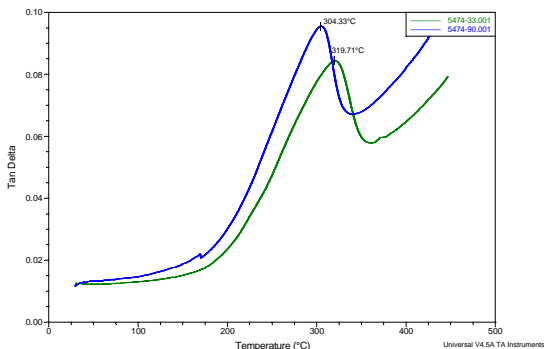


Fig. 6 $\text{Tan}\delta$ curves of the 30% and a 90% formed AlMg3 sample

Fig. 7 summarizes our results by showing the recrystallization temperatures in function of shaping grade.

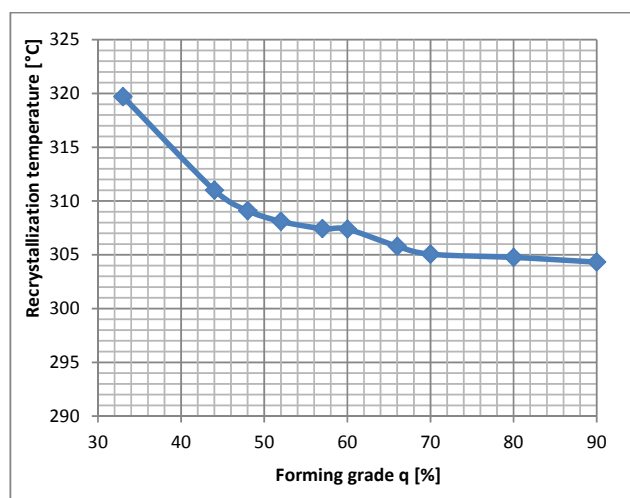


Fig. 7 Recrystallization temperatures in function of shaping grade

IV. SUMMARY

It is managed to measure the effect of cold forming on the recrystallization temperatures using DMTA equipment. The small specimen tests and the hardness tests verify the DMTA results. It can measure the recrystallization temperature of specimen with an unknown shaping grade quickly and precisely, thereby it can avoid the wrong setup of parameters.

ACKNOWLEDGMENT

This research work was performed as part of the TÁMOP-4.2.2.A-11/1/KONV-2012-0029 project in the framework of the New Hungarian Development Plan. The project is supported by the European Union, and co-financed by the European Social Fund, which is gratefully acknowledged.

REFERENCES

- [1] Damelis Pino, Luis Acuna, Ney José Luiggi: Study Of the Mg_2Si Phase in a 6063 Al Commercial Alloy Using Dilatometric Techniques, *Materials Science From Vols.* 217-222 (1996) pp 833 – 838.
- [2] G.D. Fan, M.Y. Zheng, X.S. Hu, C. Xu, K. Wu, I.S. Golovin: Effect of heat treatment on internal friction in ECAP processed commercial pure Mg, *Journal of Alloys and Compounds* 549 (2013) 38–45, 2013
- [3] G. Thomas, *J. inst. of Metals* 90, 57, 1961
- [4] A. Lutts, *Act Metall* 9, 577, 1961
- [5] R. C. Dorward, *Metall Trans* 4, 507, 1982
- [6] J. P. Linch. L. M. Brown and M. H. Jacobs *Act. Metall* 30, 1389, 1982
- [7] N. J. Luggi and A. E. Betancourt, *Metall and Mat. Trans. B* 25 B, 917, 1994
- [8] L. F. Mondolfo, *Aluminium Alloys, Structure and Properties*, Butter Worths, London 1976
- [9] Kirchfeld Mária: *Műszaki anyagok*, SZE, Győr, 2007.
- [10] Z.C. Zhou, J.Y. Xiong, S.Y. Gu, D.K. Yang, Y.J. Yan, J. Du: Anelastic relaxation caused by interstitial atoms in b-type sintered Ti–Nb alloys, *Journal of Alloys and Compounds* 509 (2011) 7356–7360, 2011
- [11] J.L. Murray, *Phase Diagram of Binary Titanium Alloys*, Materials Park, ASM, Ohio 1987.
- [12] G.D. Fan, M.Y. Zheng, X.S. Hu, C. Xu, K. Wu, I.S. Golovin: Effect of heat treatment on internal friction in ECAP processed commercial pure Mg, *Journal of Alloys and Compounds* 549 (2013) 38–45, 2013
- [13] Shih-Hang Chang: Internal friction of Cu–13.5Al–4Ni shape memory alloy measured by dynamic mechanical analysis under isothermal conditions, *Materials Letters* 64 (2010) 93–95, 2010
- [14] Adelaide Nespola, Francesca Passaretti, Elena Villa: Phase transition and mechanical damping properties: A DMTA study of NiTiCu shape memory alloys, *Intermetallics*, Volume 32 (2013) 394-400, 2013
- [15] R.E. Wetton, J.S. Fisher, K.E. Pettitt, A. Evans, J.C. Duncan, *American Laboratory*, (1993)
- [16] Menard, Kevin P.: *Dynamic Mechanical Analysis: A Practical Introduction*. CRC Press (1999)
- [17] J. Mäs et al.: "Dynamic mechanical properties of polycarbonate and acrylonitrile-butadiene-styrene copolymer blends". *Journal of Applied Polymer Science* 83 (7) (2002) 1507–1516