PLASTIC INSTABILITIES IN Al-Cu, Al-Mg AND Al-Si ALLOYS INVESTIGATED BY DYNAMIC ULTRA MICRO-HARDNESS TESTING

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Abstract

Plastic instabilities in binary high purity Al-3.3wt%Mg, Al-1.1wt%Si and Al-(0-3wt%)Cu alloys were investigated by using a Vickers hardness indenter in a dynamic ultramicrohardness testing machine. The hardness tests were performed at constant loading rates in the range of 0.7 - 70 mN/s. Load-displacement-time data were recorded during loading so that the dynamic microhardness could be calculated. The load-displacement curves obtained for the solid solution Al-Cu, Al-Mg and Al-Si alloys are not smoothly changing functions, but contain characteristic steps. This means that the dynamic microhardness oscillates quasi-periodically indicating the occurrence of plastic instabilities during indentation. The phenomenon is similar to the serrated yielding (Portevin-Le Châtelier effect) often observed also in tensile tests. In the Al-3wt%Cu the steps disappear as Guinier-Preston (GP) zones are formed during natural ageing. The occurrence and the development of the steps depend both on the loading rate and on the composition of the alloy.

Keywords: *plastic instabilities, Portevin - Le Châtelier effect, dynamic micro-hardness, Al-Cu, Al-Mg and Al-Si alloys.*

1. Introduction

Plastic instabilities as the phenomenon of repeated yielding during plastic deformation have been widely observed, and discussed in the literature under various names like Portevin-Le Châtelier (PLC) effect, serrated yielding, jerky flow etc. The phenomenon has been most often observed on metallic solid solution [1,2] alloys but it appears also in precipitation strengthened alloys in underaged condition [3]. In unidirectional deformation the most characteristic feature is the appearance of serration (stress drops or steps) beginning from a critical strain, ε_c, on the originally smooth stress-strain curves. Therefore, several works focused both theoretically [4,5] and experimentally [6] on the determination of the critical strain. The physical basis for the appearance of the PLC effect is a negative strain rate sensitivity originating mainly from the interaction between mobile dislocations and diffusing solute atoms.

The PLC effect has been observed and studied in different modes of deformation, like torsion [3], tension [7-9] etc. Recently, acoustic emission was also successfully applied to study the mechanism of the PLC effect [10]. The appearance of serration e.g. in tensile tests means that at certain points of the tensile stress-strain ($\sigma-\varepsilon$) curve the rate of work hardening, $\partial\sigma/\partial\varepsilon$ becomes negative, which physically means that sometimes the sample becomes locally softer at increasing deformation. A similar effect can be expected in depth sensitive indentation or dynamic hardness tests. First results about the occurrence and the kinematic analysis of plastic instabilities in dynamic microhardness testing have been recently reported [11,12].

2. Experimental Procedure

Binary Al-3.3wt%Mg, Al-1.1wt%Si and Al-Cu with 0.028, 0.13, 0.22, 1, 2, 3 and 4wt%Cu samples were prepared from 99.999% purity aluminium. All samples were solution heat treated for 30 minutes (for Mg and Cu solutes at 500°C and for Si at 570°C) and water-quenched to room temperature to produce a homogeneous solid solution. The ultramicrohardness measurements were made immediately after quenching on an electrolytically polished cross section of the sample. The experiments were carried out by using a Vickers hardness indenter in a dynamic ultramicrohardness testing machine (Shimadzu DUH 202). In the depth sensing hardness measurements the indentation depth (h), and the load (F) were recorded as a function of time (t) by a computer. During the tests the load is increased at a constant loading rate, μ. In the present experiments μ was changed between 0.7 and 70 *mN/s*. The load was increased up to 2000 *mN*.

3. Results and Discussion

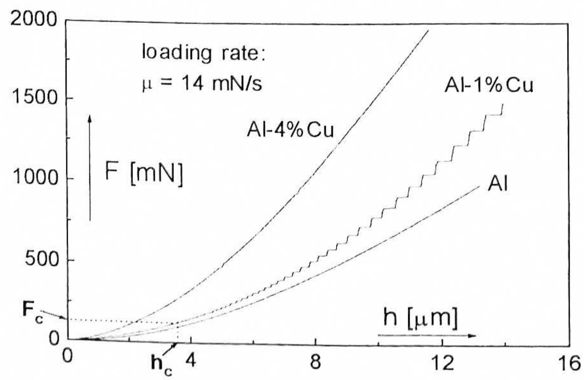

Fig. 1: Typical indentation depth-load obtained in the dynamic microhardness tests

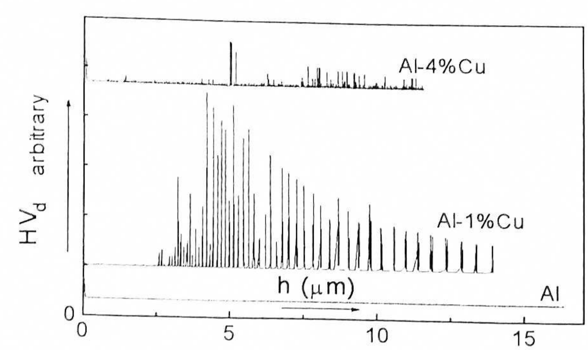

Fig. 2: Dynamic microhardness, HV_d, obtained from the F-h curves shown in Fig. 1 in the function of the indentation depth, h (The curves are shifted vertically to avoid overlapping)

Some typical depth-load curves obtained on the samples investigated are shown in Fig. 1.

Two types of the indentation depth-load curves can be distinguished: on pure Al and on the Al-4%Cu alloy smoothly changing curves were detected, while in the case of Al-1%Cu (and of some other alloys, see later) from a certain critical load, F_c (and of course from a corresponding critical depth, h_c), characteristic steps appear in the h-F curves.

To the smooth curves the quadratic

$$F = K \cdot h^2 \qquad (1)$$

function can be fitted with good accuracy where K is a constant. The occurrence and the development of the steps depend both on the loading rate and on the composition of the alloy (see it later).

To analyse the step formation process the dynamic microhardness of the sample defined as [11]:

$$HV_d = 3.8584 \cdot \left(\frac{\partial \sqrt{F}}{\partial h} \right)^2 \qquad (2)$$

can be considered. The dynamic microhardness is constant and obviously equal to the conventional Vickers-hardness for the smooth curves described by Eq.(1).

Fig. 2 shows the dynamic microhardness, HV_d, obtained from the F-h curves shown in Fig.1. It can be seen that while in the case of the smoothly changing indentation curves the value of HV_d is approximately constant, in the case of the indentation curve on which the step-like instabilities occur (for instance, for Al-1%Cu alloy) it oscillates around the conventional (static) value.

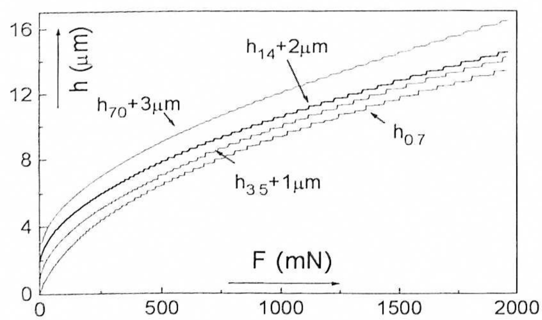

Fig.3: Indentation curves taken at different loading rates on Al-3%Mg alloy (the index means the loading rate in mN/s, the curves are shifted vertically)

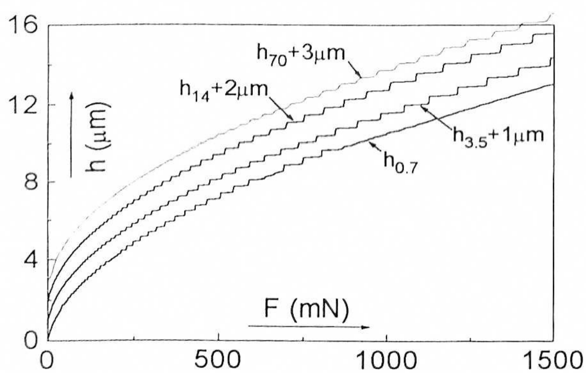

Fig. 4: Indentation curves taken at different loading rates on Al-1.1%Si alloy (the index means the loading rate in mN/s, the curves are shifted vertically)

Measurements were made on the Al-3wt%Mg alloy at different loading rates between 0.7-70 *mN/s*. Four indentation curves taken at different loading rates are shown in Fig.3. It can be seen that with decreasing loading rate, μ, the steps become sharper and the critical depth, h_c decreases.

Fig.4 shows the indentation curves obtained on the Al-1%Si alloy at four different loading rates. The instability steps at the lowest loading rate (μ=0.7 *mN/s*) disappeared in the later stage of the indentation. This may be a consequence of the strain rate dependence of the plastic instabilities [2,5,6]. For indentation tests an equivalent strain rate $\frac{1}{h}\cdot\frac{\partial h}{\partial t}$ (where t is the elapsed time during the measurement) is generally accepted [13,14], which is continuously decreasing during indentation.

In the case of Al-Cu alloy both the loading rate and the concentration dependence of the instabilities was investigated.

It is well known that in Al-Cu alloys if the Cu content (C) is higher than 2wt%, following the quenching from the supersaturated solid solution Guinier-Preston (GP) zones are formed at room temperature (RT, natural aging process) [15] which strongly change the mechanical properties of the alloy. Therefore, our investigations were extended to study the effect of natural aging, too. It should be emphasized that in the case of the Al-Mg and Al-Si alloys within about 3 months after quenching the natural aging has no effect on the occurrence and development of the steps on the indentation curves.

Fig. 5 shows some indentation depth-load curves taken at μ= 14 *mN/s* loading rate for different Cu-concentration alloys naturally aged at RT for different times, t_a.

The experimental results show that the indentation curves of the 0.028 and 0.13wt%Cu containing alloys - similarly to that of pure Al - change smoothly, no steps can be observed (also not at other loading rates). This result obviously corresponds to the fact that the physical basis of the PLC effect - the negative strain rate sensitivity - is originated mainly from the interaction between mobile dislocations and diffusing solute atoms, and a certain solute concentration is necessary.

Each curve taken immediately after quenching and polishing contains steps indicating plastic instabilities in the solid solution state of the alloys with higher (C≥ 0.22wt%) Cu concentrations (see Fig.5a). The development of the steps, however, depends strongly on the Cu-content and on the time, t_a. While in the case of the medium (0.22, 0.71 and 1wt%) Cu concentrations steps appear even after several months of t_a, on the Al-2wt%Cu alloy the instability steps disappeared after one day, and on the Al-3wt% Cu alloy after a few hours (see Figs. 5b and 5c). On the alloy with the highest Cu-concentration (Al-4%Cu) steps were not observed at all.

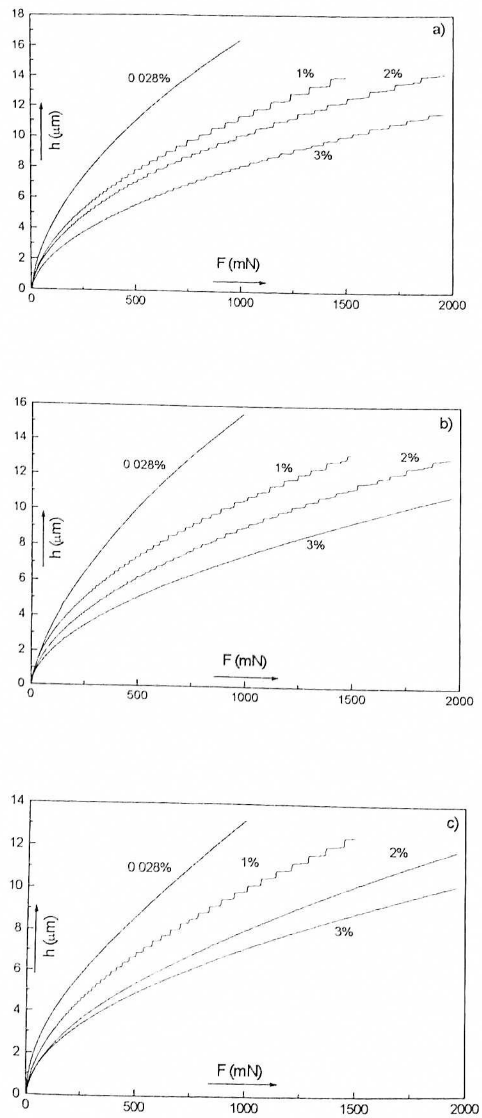

Fig. 5: The indentation curves taken at loading rate of μ= 14 mN/s on different Cu concentration Al-Cu alloys after quenching for t_a:
a) 10 minutes, b) 3 hours, c) 25 hours

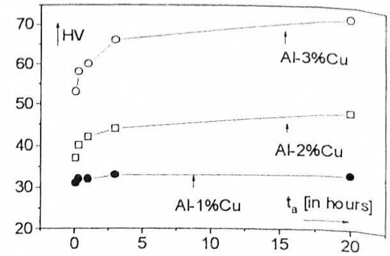

Fig. 6: Changes in the conventional Vickers microhardness (HV) of Al-1, 2 and 3%Cu alloys during the RT aging

The behaviour of the higher Cu concentration (C = 2, 3 and 4wt%) alloys can be explained by the formation of GP zones in these alloys. Fig.6 shows the changes of the conventional Vickers microhardness (HV) of the Al-Cu alloys during RT aging. While the hardness of the Al-1%Cu alloy remains practically constant with increasing t_a, the hardness of the Al-2wt%Cu and Al-3wt%Cu alloys is strongly increasing clearly showing the strengthening effect of GP zones and indicating the rapid formation of GP zones after quenching. By comparing the results of the Vickers microhardness measurements with the development of the instability steps on the indentation curves the conclusion can be drawn that the occurrence of the plastic instabilities is connected to the solid solution state of the alloys. In dilute alloys (for instance when the Cu content $C \leq 1\%$) GP zones do not form (or form very slowly), consequently these alloys remain solid solutions for long time after quenching, therefore plastic instabilities (indentation steps) were always observed. In the case of high solute content, where GP zones form rapidly the disappearance of the plastic instabilities is connected to the effect of GP zones, i.e. the interaction of dislocations with GP zones becomes dominant compared to that of the solute atoms and so the formation of GP zones suppresses the plastic instabilities, therefore the plastic instabilities disappear at a certain state of decomposition of the alloy.

To investigate the dependence of the critical indentation depth, h_c, on the solute concentration, measurements made immediately after quenching have been considered.

As reported in a previous paper [12] the most accurate method for the determination of the critical indentation depth, h_c is to investigate the $\partial h / \partial t$ indentation velocity as a function of time. The values of h_c obtained in this way as a function of the Cu-content (C) are shown in Fig. 7. It can be seen that the critical indentation depth, h_c is decreasing with increasing Cu content. To the h_c - C data shown in Fig. 7 the

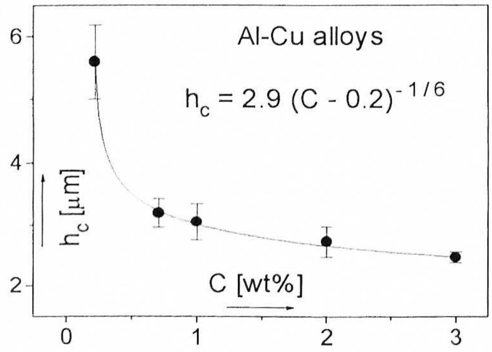

Fig. 7: The concentration dependence of the critical indentation depth, h_c

$$h_c = A \cdot (C - C_o)^n \qquad (3)$$

power-law function can be fitted with the parameters

$A = 2.90 \pm 0.13\,\mu m$,

$C_o = 0.20 \pm 0.02$ wt%

and $n = -0.16 \pm 0.05$

with which function (3) can be given approximately as

$$h_c = 2.9 \cdot (C - C_o)^{-1/6} \qquad (4)$$

The decrease of h_c with increasing Cu content is clearly a consequence of the higher number of solute atoms taking part in the dynamic strain aging effect.

According to Eq.(4) if the Cu content (C) decreases to the value of 0.2%, h_c tends to infinity. This means that at Cu contents $C \leq 0.2\%$ plastic instabilities can not occur in Al-Cu alloys. The validity of eq. (4) is confirmed by the fact that in the case of Cu content $C = 0.028$ and 0.13% plastic instabilities were not observed on the indentation curves.

4. Conclusions

Plastic instabilities in binary high purity Al-3.3wt%Mg, Al-1.1wt%Si and Al-(0-4wt%)Cu alloys were investigated by using a Vickers hardness indenter in a dynamic ultramicrohardness testing machine. The main results can be summarized as follows:

1) It was shown that the load-displacement curves obtained for the solid solution Al-Cu, Al-Mg and Al-Si alloys are not smoothly changing functions, but contain characteristic steps. This means that the dynamic micro-hardness oscillates quasi-periodically indicating the occurrence of plastic instabilities during indentation. The phenomenon is similar to the serrated yielding (Portevin-Le Châtelier effect) often observed also in tensile tests.

2) The occurrence and development of the plastic instability steps depend on the loading rate.

3) In the case of Al-Cu alloys it was shown that the plastic instabilities occur only in the solid solution state of the alloys, and it can be observed if the Cu content is larger than 0.2wt%.

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References

[1] H. Neuhäuser and C. Schwink, in *Materials Science and Technology*, eds. by R. W. Cahn, P. Haasen and E. J. Kramer, *Vol.6.* ed. by H. Mughrabi, (VCH Weinheim, Germany,1993) 191.
[2] Y. J. M. Brechet, in *Nonequilibrium Materials*, edited by J. Lendvai, Key Eng. Mater., Vol. 103. (Trans Tech Publication, Switzerland, 1995) 21.
[3] P. G. McCormic: Scripta Met., 15 (1982) 441.
[4] P. G. McCormic: Acta metall., 20 (1972) 351.
[5] L. P. Kubin and Y. Estrin: Acta metall., 38 (1990) 697.
[6] R. Král, P. Lukáč and M. Janeček: Mater. Sci. Forum, 217/222 (1996) 1025
[7] E. Pink and A. Grinberg: Acta metall., 30 (1982) 2153.
[8] L. P. Kubin, K. Chibab and Y. Estrin: Acta metall., 36 (1988) 2707.
[9] P. G. McCormic, S. Venkadesan and C. P. Ling: Scripta Met., 29 (1993) 1159.
[10] F. Chmelík, J. Balík, P. Lukáč, E. Pink and M. Čepová: Mater. Sci. Forum, 217/222 (1996) 1019.
[11] Gy. Bérces, N. Q. Chinh, A. Juhász and J. Lendvai: J. Mater. Res., to be published, (1998)
[12] Gy. Bérces, N. Q. Chinh, A. Juhász and J. Lendvai: Acta mater., to be published, (1998)
[13] M. S. Bobji and S. K. Biswas: *Strain rate response of materials from depth sensing indentation* in "Recent Advances in Metallurgical Processes", (New Age Int. Publishers, New Delhi, 1997) 1.
[14] W. H. Poisl, W. C. Oliver and B. D. Fabes: J. Mater. Res. 10 (1995) 2024.
[15] R. W. Cahn, *Physical Metallurgy*, American Elsevier Publishing Co. Inc., New York, 1970.