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Mechanical properties and microstructure of laser treated Al-Cu-Mg alloys

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Abstract- The mechanical properties and microstructural features of Al-Cu-Mg alloys were investigated, as exposed to laser treatments at various scan velocities. As far as the mechanical property is concerned a striking observation is a minimum in the hardness value at a laser scan velocity of 1/2 cm/s. Usually an increasing hardness with increasing laser scan velocities is reported in the literature. This remarkable property could be explained based on the microstructural features observed by transmission electron microscopy.

After subsequent shot peening, in all cases the formation of precipitates was observed, independent of the laser scan velocities originally applied. This phenomenon of precipitation, induced by shot peening afterwards is most striking at a high concentration of alloying elements in solid solution. Since this is the case in samples treated at a low scan velocity (i.e. an increased homogenization time) as well as in samples treated at a high scan velocity samples (high quench rates), the parabola shape is maintained even after shot peening.

1. INTRODUCTION.

Among the available laser applications laser surface melting has turned out to be a powerful technique for the production of wear resistant layers [1]. Despite the advantages of this process, laser surface melting results in tensile stresses which may assist crack propagation. It will be shown that shot peening can overcome this drawback effectively.

In our previous work we concentrated on the combination laser-shot peening treatment in the case of an eutectic aluminium-silicon alloy [2]. It turned out that a laser treatment of a cast aluminium-silicium alloy leads to a very fine dispersion of the silicon phase in the Al-matrix. In addition, it was shown that the laser treatment amplifies substantially the effectiveness of a subsequent shot peening treatment. In particular the maximum attainable hardness and compressive stress turned out to increase upon increasing quench rate, i.e. upon increasing the laser scan velocity. The appearance of shot peening induced precipitates forms the most striking observation in that study.

This paper concentrates on the mechanical properties and microstructural features of Al-Cu-Mg alloys exposed to laser treatments at various scan velocities followed by shot peening treatments. Aluminium alloys containing Cu, Mg and small weight percentages of other alloying elements have shown to develop high strength after appropriate heat treatments. Nevertheless improvement of the mechanical properties along conventional processing routes has its limits, and alternative techniques like splat cooling and laser melting are increasingly being explored. Conventional hardening treatments of aluminium-base alloys usually exploits the beneficial effects of a high concentration of precipitates.

In contrast, hardening by laser treatments is achieved primarily by the small grain sizes that are formed as a result of the rapid solidification process. The flow stress varies as $d^{-\alpha}$ where α depends on the cell size and cell wall-type. Since the cell size is inversely related to the solidification rate, commonly an increasing hardness is observed with increasing laser scan velocity.

2. EXPERIMENTS.

Samples of commercial Al 2024-T3 (4.4% Cu, 1.5% Mg, 0.6% Mn and Bal. Al.; solution treated, cold rolled and naturally aged) were sandblasted to obtain a rough well absorbing surface. After ultrasonically cleaning the samples were irradiated using a transverse flow Spectra Physics 820 CW-CO_2 laser under a protective argon atmosphere. At the surface the power of the beam was 1300 W. The focus point of the ZnSe lens with focal length of 127 mm lay 5 mm above the surface, resulting in a spot diameter of 0.75 mm. Single tracks were made at laser scan velocities ranging between 1/8 to 25 cm/s. On the samples for analysis by X-ray diffraction several adjacent tracks were laid down.

It should be emphasized that of all the elements in Al 2024, magnesium has by far the lowest boiling point (1390 K), and it is therefore to be expected that a substantial fraction of magnesium will actually evaporate.

Shot peening was carried out in a conventional blast cleaning apparatus, applying glass beads with an average diameter of 720 µm. The air pressure was 2.6 bar. Vickers hardness measurements were carried out just below the surface of a taper sectioned sample, with a 100 gram weight. TEM samples were prepared using a mechanical dimpler, followed by ion milling in the cold stage of a Gatan ion mill, in such a way that the electron transparent area is located in the centre of the laser track at a depth of approximately 30 µm. The hardness measurements provide information of this area as well.

X-ray analysis applying Cu-radiation, revealed the stress state after laser melting and subsequent shot peening, utilising the <422> reflection. Line profile analyses on the <111> reflection were carried out to obtain information about the dislocation density. For further experimental details we refer to [2].

3. RESULTS.

After laser melting a cellular structure develops during solidification. Cracks are occasionally observed in samples treated at low as well as at high laser scan velocity, indicating a reduced ductility at both ends of the scan velocity range. The measured cell sizes are depicted in Fig. 1. The intermetallic compounds mainly consist of the $CuAl_2$ (θ) phase, and the $CuMgAl_2$ (S) phase. These phases were identified with by X-ray diff?raction.

Because of the rapid hardening right after the laser treatment, all hardness measurements presented in the following are performed on samples aged for at least two weeks. The surface hardness as a function of laser scan velocity, is displayed in Fig. 2. This graph exhibits a parabolic shape, with a minimum value at a scan velocity of 1/2 cm/s. After subsequent shot peening the shape of the hardness curve remains approximately the same, although it shifts to higher values.

TEM specimens taken from samples exposed to different laser scan velocity samples before shot peening show marked differences in microstructural features. The samples treated at a lower scan velocity, 1/8 cm/s to 1/2 cm/s, are mainly precipitate free although in some areas coarser precipitates could be observed. However, it is uncertain whether these are partly dissolved cell boundaries or real precipitates.

The most striking feature is the formation of helical dislocations as being observed in all samples treated at low laser scan velocity. Most of these dislocations exhibit a uniform equilibrium shape with respect to pitch and radius. At the lowest scan velocity however, non uniform shapes are occasionally observed.

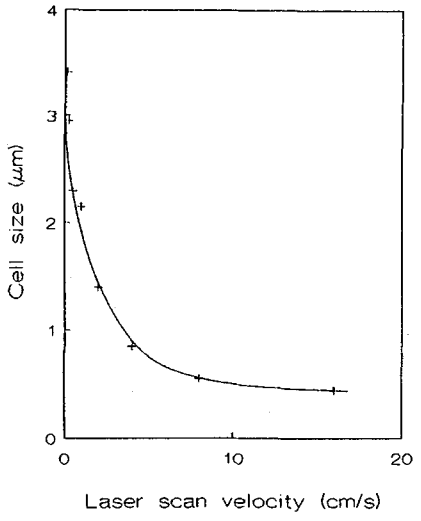

Fig 1 Cell size as a function of laser scan velocity.

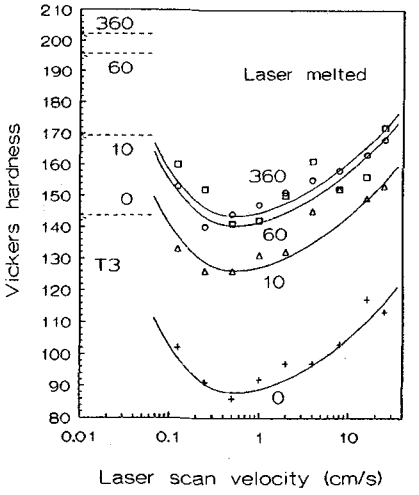

Fig 2 Hardness profile as a function of laser scan velocity and shot peen time (s).

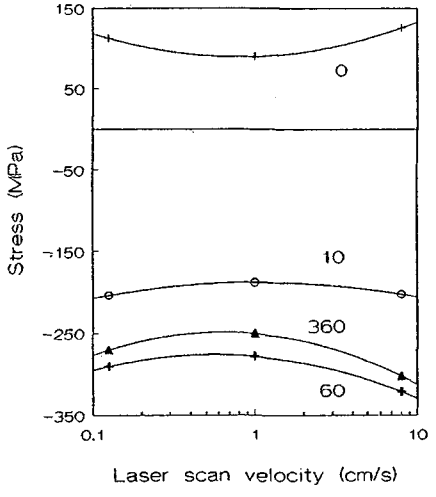

Fig 3 Residual stress as a function of laser scan velocity and shot peen time.

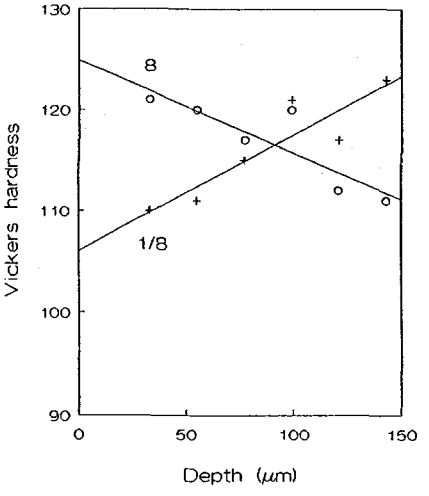

Fig 4 Hardness profile as a function of depth of two samples treated at a low and a high laser scan velocity.

In samples treated at laser scan velocities between 1 cm/s to 4 cm/s the precipitation of the tetragonal θ' platelets, with an average size of 150 nm, is observed. Since these platelets are oriented parallel to the {100} faces of the α Al, we may conclude that these precipitates are not the so-called S' precipitates (Al_2CuMg). The S' precipitates grow locally on {120} planes, giving {110}, {100} or {130} as an overall growth plane [3][4]. Beside the platelets a more globular precipitate is also observed in these samples, mainly nucleating on the edges of the θ' plates and on dislocations. This could be the θ phase as well as the S' phase. Since in alloys with a copper:magnesium ratio less than 8:1 Al_2CuMg precipitates can start to play a role [5], the existence of the S and S' phase cannot be excluded. Electron diffraction patterns have not yet revealed the nature of these precipitates.

Samples treated at laser scan velocities of 8 cm/s to 25 cm/s show only a cellular structure, in which it is seen that the cell boundaries act as obstacles for dislocations motion. GP-zones, that could precede the formation of the θ' plates, are not observed.

After shot peening, in all samples a higher dislocation density was observed by TEM. Apart from the increased dislocation density however, now precipitates could be detected in samples that did not develop observable precipitates before shot peening. A typical example of this precipitation process induced by shot peening in a sample treated at a low laser scan velocity of 1/8 cm/s. The concentration of precipitates seems also to be higher than before peening in a samples treated in the scan velocity range of 1 cm/s to 4 cm/s. This might indicate an increased solid solution in these samples, but the smaller average size, 100 nm, will also result in a higher concentration. Again, most of these precipitates are θ'-platelets, aligned with the cubic axes. A second type of precipitate, smaller in size but also of the shape of a platelet, is identified as the S' phase, initially growing on [120] planes. These precipitates have a much smaller width-to-thickness ratio and will therefore contribute less to an increase of the flow stress than the θ'-plates (at the same volume fraction). The very broad size distribution, down to only 2 nm, makes this contribution more uncertain.

X-ray stress measurements reveal a similar parabola shaped curve for the residual stress state, as is displayed in Fig. 3. Before shot peening the stress state has a tensile character with an average magnitude of 100 MPa. After shot peening the stress is inverted to a compressive one with a magnitude in the order of 250 MPa, while maintaining the parabola shape. The magnitude of the stress after 360 s shot peening is lower than after 60 s. This is not due to an inaccuracy of the measurement. In fact it turned out to be reproducible by peening the 60 s sample for an extra 300 s period.

Line profile analysis was applied to obtain information on the dislocation density [6]. This shows again a minimum as a function of laser scan velocity. The increase in dislocation density is approximately the same for all different samples except for the decrease after 360 s of shot peening of samples treated at a high scan velocity.

4. DISCUSSION AND ANALYSIS.

To discuss the experimental results, the laser scan velocity range is divided into two regimes: the low scan velocity regime (1/8 cm/s to 1/2 cm/s) and the high scan velocity regime (1/2 cm/s to 25 cm/s).

low scan velocity regime

Since the cooling rates are slower at the low laser scan velocity, compared to the 2 cm/s, the latter of which precipitation of the θ' plates is observed, at first sight one would expect to see an increasing nucleation an growth of precipitates in these samples. In contrast, below 1 cm/s no significant precipitation is observed. The explanation for this observation lies in the decreasing vacancy concentration with decreasing laser scan velocity. Apparently at the lower laser scan velocity the vacancies are annealed out, a.o. by absorption on dislocations that causes the formation of helical

dislocations, and before the temperature reaches values that would favour the nucleation and growth of observable precipitates. Therefore the rise in hardness values in the low velocity regime can not be caused by precipitation hardening but due to two other contributions:

First, the dissolution of the cell-walls will play an important role. Since there exists only a small temperature interval during which homogenization takes place [5], the cooling rate must be low enough to allow sufficient time for diffusion. The copper (and magnesium) brought into solid solution will contribute to an enhanced hardness but for reasons described above, copper will not form any precipitate. The dissolution may continue as long as not all cell walls have been fully dissolved, provided that the cooling rate is low enough. Obviously this situation has not been reached in practice, and it explains why the hardness is still increasing with decreasing laser scan velocity. In some additional experiments performed on an alloy with 2.32 w% Cu, it was observed that only near the edges of the low scan velocity laser tracks the cell walls were completely dissolved. From this experiment we conclude that to a first approximation only half of the copper is in solid solution in Al2024 treated with a scan velocity of 1/8 cm/s. A calculation of the diffusion distance in a 1/8 cm/s track, during cooling in the interval between 660 °C and 450 °C, applying an analytical model [7] yields a distance of approximately 0.1 μm. A calculation for magnesium yields a similar value. These value, that should only be regarded as a first approximation, are too small for significant homogenization. Nevertheless, it shows that homogenization may play a role only at the lowest scan velocities.

An indication that supports homogenization as being the responsible mechanism for the hardness increase in the low scan velocity regime, lies in the observation that in a laser track of 1/8 cm/s scan velocity the hardness increases with depth, as displayed in Fig. 4. This is a result of the increased homogenization time with increasing depths. In laser tracks where this mechanism does not operate, e.g. a 8 cm/s track, hardness decrease is detected with increasing depth. This decrease is also observed in other materials [2] where indeed homogenization is not a likely process to occur.

Beside homogenization, another contribution to the increased hardness in the low scan velocity regime concerns the formation of helical dislocations. Since glide of a helical dislocation is restricted to its cylindrical surface on which it is wound, these dislocation are essentially immobile. The formation of helical dislocations is observed in all samples treated at low laser scan velocity. The large quantity of vacancies is present because of the substantial cooling rates from high temperatures. The reason that the formation is possible only in samples treated at the low laser scan velocity is that only here the cooling rate at intermediate temperatures is low enough to allow for adequate vacancy diffusion. Once the temperature is too low for significant vacancy diffusion, the dislocations become immobile over the distance that has been affected by climb or by the interaction with the vacancy loop. The equilibrium shape of a helical dislocation is one of a constant pitch and loop radius [8]. Since a non-equilibrium shape is sometimes observed, we conclude that in the sample treated at the lowest scan velocity the anneal time is still not long enough for all the dislocations to reach this equilibrium situation. This in turn can be caused by the continuous formation of new helical segments, which means that not all vacancies are used, or by an increased locked segment through relaxation to the equilibrium shape. These explanations are also in line with the fact that the hardness curve is still rising at the 1/8 cm/s point. Nevertheless we do not expect that this mechanism will dominate over the role of homogenization as described first.

high scan velocity regime

The increased hardness in the high velocity regime, reflects the general observed trend, and is caused by two reasons. First, the cell size decreases upon increasing the laser scan velocity and causes a hardening. Furthermore, the observed precipitates will contribute to the hardness values, and when this contribution gets large the contribution arising from the small cell size may become negligible. The presence of these precipitates is observed in samples with scan velocities of 1 cm/s to 4 cm/s.

The absence of GP zones in samples that have not yet developed precipitates, in the range between 8 cm/s and 25 cm/s, might be explained by the fact that the amount of Cu in solid solution is less compared to conventionally heat treated Al 2024. In Al with 2 w% Cu, GP-I zones do not appear, and the precipitation sequence starts with GP-II [5]. Furthermore it has been reported that in rapidly quenched samples the early stages in the precipitation sequence are accelerated and that GP-II zone formation is suppressed in favour of the θ′ phase. If both effects are present in our samples the θ′ phase is indeed the first precipitate to be observed.

After shot peening an increase in hardness that is independent of the laser scan velocity is observed. This is not what one would expect since the microstructure is quite different at different scan velocities. The precipitation induced by shot peening, that was observed earlier in a laser melted Al-Si alloy [2], explains why the observed values are the same for the low and high scan velocities: After shot peening precipitates are present independent of the scan velocity regimes, whereas the amount of alloying elements in solid solution is expected to be much less than before shot peening [1][2].

The minimum value of the hardness of samples treated at a scan velocity of 1/2 cm/s is still present after shot peening, and is also visible in the curves of the residual stresses as well as of the dislocation density vs laser scan velocity. Apparently the amount alloying elements brought into solid solution during laser melting has a minimum for this scan velocity. At higher scan velocities more copper and magnesium is retained during the rapid solidification process, and for the lower ones the homogenization time during cooling is longer.

5. CONCLUSIONS.

The hardness curve observed in this study, with a minimum at a laser scan velocity of 1/2 cm/s, is quite different from the monotonically increasing curves, usually detected in many other laser treated materials. The reason for this observation is the minimum in the solid solution concentration. By varying the quench rate it is possible to achieve different stages in the precipitation sequence.

The mechanisms by which the hardening occurs are the same as the ones in conventionally hardened specimens, except for the contributions originating from the small cell sizes and from the formation of helical dislocations. The latter ones however do not seem to contribute significantly.

Shotpeening leads to a further hardness increase, which can be attributed to the creation of (additional) θ′ and S' precipitates. An increased dislocation density contributes to the increased hardness as well.

references

[1] De Beurs, H. and De Hosson, J.Th.M, High power lasers (Eds. A. Niku-Lari and B. L. Mordike) Pergamon Press, Oxford, **27** (1988).

[2] Noordhuis, J., De Hosson, J.Th.M., Acta Metall et Mat. **40** (1992) 3317.

[3] Wilson, R.N., Partridge, P.G., Acta Metall., **13** (1965) 1321.

[4] Stolz, R.E., Pelloux, R.M., Met. Trans., **7A** (1976) 1295.

[5] Mondolfo, L.F., Aluminum alloys structure and properties, (1976) 264.

[6] Williamson, G.K., Smallman R.E., Philos. Mag., **1** (1956) 34.

[7] Ashby, M.F., Easterling, K.E., Acta Metall., **32** (1984) 1935.

[8] Hirth, J.P., Lothe, J., Theory of dislocations, McGraw Hill, New York, 1982, 623.