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MICROSTRUCTURE OF LASER TREATED Al ALLOYS

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Abstract—In this study the microstructures of laser treated ultra pure Al and two Al–Si alloys (Al–0.4 Si and Al–0.75 Si) were investigated. In ultra pure Al a large number of dislocation loops were found especially at higher laser scan velocities. During annealing only at laser scan velocities above 2 cm/s a large quantity of dislocation loops became visible. Both results indicate that at high laser velocities vacancies are frozen in, but at laser velocities around 1 cm/s there is still enough time at high temperature to reduce the vacancy concentration towards lower super-saturation. In Al–Si alloys the dislocation density rises with higher laser scan velocities probably caused by the smaller distances between the eutectic cell walls. In these alloys entangled dislocation structures were found in contrast to ultra pure Al. For solidification structures consisting of an eutectic material with a high hardness as for an eutectic structure in Al–Si alloys it was found that the hardness can be described by a pile up mechanism, which depends on the difficulty to exert stresses on neighbouring cells due to thick and hard walls. The hardness has been described by a $1/d^2$ dependence, i.e. it is mainly determined by the small size d , of the solidification structure.

Résumé—Dans ce travail, on étudie les microstructures d'aluminium ultra-pur et de deux alliages Al–Si (Al–0,4 Si et Al–0,75 Si) traités au laser. Dans l'aluminium ultra-pur un grand nombre de boucles de dislocations sont observées, spécialement pour des vitesses de balayage élevées du laser. Pendant le recuit, et seulement pour des vitesses de balayage supérieures à 2 cm/s une grande quantité de boucles de dislocations devient visible. Ces deux résultats indiquent que, pour des vitesses élevées de balayage du laser, les lacunes sont gelées, mais qu'à des vitesses de l'ordre de 1 cm/s il y a encore assez de temps à haute température pour abaisser la concentration en lacunes vers une sursaturation moindre. Dans les alliages Al–Si, la densité de dislocations augmente pour les vitesses de balayage les plus élevées, sans doute à cause des plus faibles distances entre les parois des cellules de l'eutectique. Dans ces alliages, on n'observe pas de structure de dislocations enchevêtrées comme celles de l'aluminium ultra-pur. Pour les structures de solidification formées d'un matériau eutectique de haute dureté, aussi bien que pour une structure eutectique dans les alliages Al–Si, on trouve que la dureté peut être décrite par un mécanisme d'empilement qui dépend de la difficulté à exercer des contraintes sur les cellules avoisinantes par suite de l'épaisseur et de la dureté des parois. La dureté est décrite par une fonction en $1/d^2$, c'est à dire qu'elle est principalement déterminée par la petite taille, d , de la structure de solidification.

Zusammenfassung—In dieser Arbeit wird die durch Laserbestrahlung induzierte Mikrostruktur an ultrareinem Al und an zwei Al–Si–Legierungen (Al–0,4 Si und Al–0,75 Si) untersucht. In ultrareinem Al werden eine große Zahl von Versetzungsringen, insbesondere nach Laser-Bestrahlung mit höherer Rastergeschwindigkeit, gefunden. Während des Ausheilvorganges bleibt ein große Menge an Versetzungsringen zurück, wenn die Rastergeschwindigkeit oberhalb von 2 cm/s liegt. Beide Ergebnisse bedeuten, daß die Leerstellen bei hoher Rastergeschwindigkeit eingefroren werden, bei einer Rastergeschwindigkeit um 1 cm/s jedoch genügend Zeit bei hoher Temperatur verbleibt, um die Leerstellenkonzentration zu kleineren Übersättigungen hin zu verändern. Bei Al–Si steigt die Versetzungsdichte mit höherer Rastergeschwindigkeit, wahrscheinlich verursacht durch die kleineren Abstände zwischen den eutektischen Zellwänden. In diesen Legierungen wurden Versetzungsknäuel—im Gegensatz zu reinem Al—nicht gefunden. Für Erstarrungsstrukturen, die aus einem eutektischen Material mit großer Härte bestehen, wie etwa die eutektische Struktur in den Al–Si–Legierungen, ergibt sich, daß die Härte mit einem Aufstaumechanismus beschrieben werden kann, der von der Schwierigkeit abhängt, Spannungen wegen der dicken und harten Wände auf die benachbarten Zellen auszuüben. Die Härte läßt sich mit einer $1/d^2$ -Abhängigkeit beschreiben, d.h. sie ist überwiegend bestimmt durch die kleine Größe d der Erstarrungsstruktur.

1. INTRODUCTION

At present a high power laser is more frequently used in the field of surface engineering. Primarily its high local energy input at the surface and the high velocity of the beam are the most important advantages. The most promising potential applications of laser applied coating are small areas as, for example, cutting edges, edges of valves in engines and on scrapers.

Various methods exist to use a laser in material processing. During the passage of a laser beam the substrate material may be rapidly heated and in most applications melted. After passing of the beam the material solidifies rapidly and is quenched due to conduction of heat to the deeper parts of the substrate, which act as a heat sink. A melted and resolidified area remains embedded in a heat affected zone both in the form of the lower part of an ellipse.

In wear resistant applications it is important to have some insight in the mechanical and micro-structural aspects in relation with the laser treatment. During the treatment residual stresses and cracks may develop because of inhomogeneous thermal expansion and shrinkage. In particular dislocation structures are formed during the cooling process and vacancy concentrations can be maintained at a high, non-equilibrium level affecting e.g. diffusion and creep.

In general, laser melted and re-solidified materials possess a solidification structure which consists of a large number of cells or dendrites, separated by small angle grain boundaries or by areas of eutectic material. In contrast to more conventional solidification structures, after laser treatment a very fine structure is developed with cell sizes in the order of a few μm , i.e. comparable to the dislocation cells produced by plastic deformation. In this study the microstructural feature of laser treated pure Al and two AlSi alloys were investigated.

In addition, the hardness is studied because it gives an indication for plastic yielding properties. As a matter of fact, hardness is not the only parameter for wear, but it often gives a good indication for the wear rate of two comparable wear systems. From a more fundamental point of view it is interesting to study the relation between hardness and dislocation behaviour in laser treated material because its fine micro-structure hinders dislocation movement.

2. EXPERIMENTAL

In this study ultrapure Al and two Al-Si alloys (Al-4.0 Si and Al-7.5 Si) are investigated. The specimens were ground and sand blasted to get a rough surface, which absorbed well the laser light used (wavelength $10.6\ \mu\text{m}$). After sand blasting the samples were ultrasonically cleaned. The specimens were mounted on a rotating table and irradiated by a 1.5 kW CO_2 Spectra Physics 820 laser. The Gaussian beam was deflected by a Mo mirror and focussed by a ZnSe lens before it impinges on the surface. At the surface the power of the beam was 1300 W. The focus point of the lens lay 5 mm below the surface. The spot size of the laser beam at the surface is 0.40 mm and the depth of the trace is about 0.2 mm. A separation of the different passes of approximately 5 mm provided that there was no effect of adjacent passes by the next one. The scan velocities used were between 1 and 40 cm/s.

After the laser treatment cross-sectional samples were prepared for hardness measurement and optical (OM) and scanning electron microscopy (SEM). Microscopic studies were performed to measure the cellular/dendritical widths and the percentage wall area vs cell area using a point counting method to determine the cell wall ratio. Vickers hardness measurements were made with a weight of 0.5 N for Al and the Al-Si alloys on the cross-sections. The indentations were placed on a row parallel to

the surface and at least 1.5 times the length of the diagonal below it to get reliable measurements. Transmission electron microscopy (TEM) samples were made by taking 3 mm disks (thickness 0.2 mm) parallel and just below the surface and thinning them electrochemically until transparent area was attained. TEM samples were taken out of the middle of the laser trace.

For the determination of the dislocation density the exit method was used [1]. TEM photographs were taken using a dark field weak beam method applying a $\langle 220 \rangle$ diffraction vector to resolve $1/2 \langle 110 \rangle$ dislocations. The dislocation density was measured by counting the exits at the surface of the sample.

3. EXPERIMENTAL RESULTS

3.1. Dislocation microstructure and defect loops

In ultra pure Al a relatively low dislocation density was found. No large variation with laser velocity was detected (Fig. 1). Subgrain boundaries consisting of

Fig. 1. Dislocation densities in pure Al, Al-4 Si and Al-7.5 Si.

Fig. 2. Dislocation structure in pure Al, scan velocity laser 1 cm/s.

Fig. 3. Cross section of pure Al with laser pass (laser velocity 5 cm/s).

Fig. 4. Distribution of dislocations and vacancy loops near a subgrain boundary in pure Al (laser scan velocity 20 cm/s).

dislocation structures were formed inhomogeneously throughout the laser melted area (Fig. 2). Sometimes entangled dislocation structures and pile-ups were observed. Optical and scanning electron microscopy revealed semicircular etching structures, along with the solidification front, which consisted of a large number of etch pits (Fig. 3). It has to be noted that the ring pattern observed may be due to convection produced by the Marangoni effect and not due to shrinkage effects. At higher solidification velocities dislocation loops could be detected. No evidence for re-crystallization was found.

The microstructure of laser treated material of the Al-alloys exhibits dendrites or cells divided by an eutectic structure with very fine Al and Si platelets. Between the different cells small orientation differences were observed. A homogeneous distribution of dislocations in cells was detected. Some dislocations were connected with the eutectic walls. Other dislocations are distributed randomly over the cell interior. The dislocation density rises for both alloys with laser scan velocity. In Al-4 Si the slope is a little steeper than in Al-7.5 Si although the differences lie almost within the experimental error (Fig. 1).

The determination of vacancy concentrations in laser treated material by e.g. resistivity measurements, is rather difficult due to the heterogeneous structures. However, defect loops could be used as indirect evidence of quenched in vacancies during the examination by TEM of laser treated pure Al(99.999%). The time between laser treatment and examination of the samples was kept the same to avoid differences due to various aging times.

The maximum size of the loops was about 100 nm independent of laser scan velocity. In areas with a lower density of loops the average size seemed to be larger than in areas with a higher concentration; however, these differences are relatively small (in order of tens of nm). The concentration of loops varies strongly over the area inspected. In the neighbourhood of subgrain boundaries a depleted area was found (Fig. 4). Most loops were located in areas with a low

dislocation density. The average density of defect loops increases with increasing laser scan velocity (Fig. 5). In some TEM photographs of Al-4 Si and Al-7.5 Si alloys a few dislocation loops could be found after laser treatment. But on the whole no significant numbers of loops were formed in the Al-Si alloys studied.

Laser treated pure Al was annealed *in situ* in a transmission electron microscope. In 20 min time the temperature was slowly increased from room temperature to 553 K and kept constant at this temperature for 30 min. Independent of laser velocity annealing of dislocations was observed around a temperature of 473 K.

After laser treatment with a scan velocity of 1 cm/s no defect loops were formed during annealing whereas a laser treatment of 2 cm/s resulted in the nucleation and growth of some vacancy loops. During annealing

Fig. 5. Dislocation loop density in laser treated Al vs laser scan velocity.

Fig. 6. Average distance between dislocation loops which are formed during annealing vs laser scan velocity.

of samples produced with higher scan velocities a large number of loops were formed. The temperature at which loops became visible lies around 490 K. The distance between the loops as a function of the laser velocity is depicted in Fig. 6.

3.2. Hardness measurements

Al-Si alloys. Aluminium with 4 wt% and 7.5 wt% Si were used for hardness measurement. Values for the average hardness in a laser pass are depicted in Fig. 7. Within the velocity range used here the volume of the eutectic wall structure was independent of the velocity. For the Al-4 wt% Si alloy it was $30 \pm 3\%$, for the Al-7.5 wt% Si alloy it was $39 \pm 9\%$. In Fig. 8 the hardness is displayed vs the inverse of the average obstacle distance squared (cell/dendrite size), giving a linear correlation as can also be derived theoretically.

Fig. 8. (a) Hardness vs $1/d^2$ in Al-4 Si. (b) Hardness vs $1/d^2$ in Al-7.5 Si.

4. DISCUSSION

Dislocations are generated in the laser treated Al-materials due to the shrinkage that may for the

Fig. 7. Hardness vs laser scan velocity in Al-Si alloys.

larger part be accomplished in the melt zone. As a result a ring-like pattern consisting of etch pits is observed, with at the surface solidification dimples with an amplitude of a few micrometers. In the literature they are ascribed to small discontinuities in the laser beam, but they may well be the effect of stresses occurring during solidification.

During cooling of the samples some thermal strain should be compensated for. Because at high temperatures the yield stress is low, plastic deformation occurs easily. Therefore, one can state that the deformation rate (dislocation density) is to first order equivalent to the thermal shrinkage

$$\dot{\epsilon} = \alpha \cdot \frac{dT}{dt} \quad (1)$$

α = linear thermal expansion. This yields for a laser treatment in pure Al at temperatures between the melting point and 600 K deformation rates of $0.1\text{--}1\text{ s}^{-1}$ during e.g. $\pm 50\text{ ms}$, giving still rather small deformations in total.

At high temperatures different processes are active during deformation. Apart from glide of dislocations it is also possible that dislocations climb by diffusion or cross slip under influence of a stress field.

In pure Al almost a constant dislocation density was found independent of laser velocity. Only during the first part of the cooling curve plastic deformation occurs; at decreasing temperature the yield stress rises more rapidly than the Young's modulus. As a result elastic strain can take up thermal stresses. During this period recovery must have been occurred by which apparently differences in density are annealed out. By a combination of cross-slip and climb dislocations move to other slip planes and form subgrain boundaries or annihilate. Although the time for these processes is short it is long enough to allow diffusion on a microstructural scale as can be checked from the diffusion distances. The self diffusion distance for Al lies in the order of a micrometer for laser velocities ranging between 1 and 20 cm/s. The dislocation structures formed are comparable to those formed during isothermal deformation at 723 K with a strain rate of 0.18 s^{-1} [2].

In Al-Si alloys the dislocation density rises with laser scan velocity. In this case during deformation movement and annealing of dislocations is severely hindered by obstacles e.g. eutectic cell walls. In Al-Si alloys with small cells surrounded by hard walls a larger number of dislocations are necessary if the cells are smaller. Although this model is strictly derived for low temperatures, other work on particle hardened materials indicates that there is an inverse relationship between strain rate (and dislocation density) and distance between obstacles until fairly high temperatures [3]. No entangled structures are found in these alloys probably due to the small cell size.

At the melting point of Al the vacancy concentration is 0.09 at.% vacant lattice points. If the material is slowly cooled the vacancy concentration

Fig. 9. Diffusion distances of monovacancies in Al during a laser treatment (absorption 0.2).

adapt itself continuously to the equilibrium concentration and the concentration falls off exponentially [4]. At low temperatures clusters form [5]. Already within seconds clustering occurs at 275 K. Growth to larger sized clusters takes much longer times [6]. The clusters may form platelets which can be regarded as intrinsic stacking faults bound by Frank sessile dislocations. Due to the high stacking fault energy in Al these faulted loops rapidly unfault by a Shockley partial forming a glissile dislocation loop.

Using the Ashby-Easterling model [7] we calculated diffusion distances of mono-vacancies during a laser treatment using the same parameters as in the experiment and an absorption of 0.2 (Fig. 9). The value taken for the absorption, 0.2, has to be considered as an upper limit. Upon taking a variable value between 0.03 at scan velocity 1 cm/s and 0.2 at scan velocity 20 cm/s it turned out that the diffusion distances are somewhat smaller at lower values of the scan velocities than at a constant value of absorption 0.2. Especially at low laser scan velocities large diffusion distances are found. Di-vacancies are migrating even faster. Probably most of the vacancies have already migrated to sinks like grain boundaries before at lower temperatures loops can be formed (Fig. 2). Based on the average size observed ($\pm 50\text{ nm}$) we estimate that the defect loops contain only 1% of the equilibrium melting point concentration of vacancies. Therefore, we have observed dislocation loops in Al nucleated at lower temperatures [8] which are achieved faster in a treatment with high scan velocities. This is supported by [9]: after a pulsed laser treatment (pulse duration in the order of 15 ns) of Al also dislocation loops are found indicating that loop nucleation occurs readily.

In the annealing experiments only the samples treated with higher laser velocities showed loop formation. It was not clear whether these loops were nucleated and grown at the annealing temperature or already nucleated during the laser cooling cycle and grown to visible sizes during annealing. In both cases clusters

do not shrink but grow despite the rise in temperature which causes dislocations and other sources normally to emit vacancies to maintain equilibrium with the result that dislocation loops shrink.

In Al-Si almost no dislocation loops are formed because here more sinks are present in the form of phase boundaries and dislocations associated with them. Figure 8 shows a $1/d^2$ dependence of the hardness (d is the cell size) that is in contrast to the commonly found Hall-Petch relationship [10]. This can be explained as follows. In the laser melted materials used here a structure is developed during re-solidification of relatively small dendrites or cells, in some cases surrounded by hard walls. At the yield point the hard walls can be seen as pinning point and neglecting the effect of other dislocations the minimum stress τ_c to move the dislocation is the Orowan stress [11].

When these dislocations are pinned and further plastic deformation is enforced it is necessary that new dislocations are generated. So, dislocation sources must be activated. At a low dislocation density a source (e.g. Frank-Read source) can emit dislocations without being influenced much by the emitted dislocations.

The Frank-Read stress τ_{FR} , the stress necessary to bow out a dislocation between a source length AB , is given by

$$\tau_{FR} = \tau_0 + \frac{CGb}{2\pi AB} \ln\left(\frac{AB}{b}\right) \quad (2)$$

where b is the magnitude of the Burgers vector, G the shear modulus and C a constant. τ_0 represents the friction stress on dislocations which is considered to be due to all causes except interactions with the elastic stress fields of other dislocations. In the following it is assumed that the dislocation source lies in the center of the cell and possesses a source length $AB = 1/2d$. Because in laser treated materials the cells are very small (size $\approx 1 \mu\text{m}$), i.e. too small for development of a dislocation cell structure, the dislocations are stopped by the cell wall and a pile up is formed. This pile up exerts a back stress on the dislocation source. To find a relation between stress and strain we assume that all the cells have the same yield stress τ_1 [12]. Because the different cells have different orientations, they have different resolved shear stresses $\tau_i = \tau/m_i$ ($m_i > 1$) for the i th cell. The i th cell starts yielding when τ_i becomes larger than τ_1 . Its Frank-Read source will emit loops, which pile up against cell walls. Because the cell walls are relatively thick they are hard to penetrate. There are only a few sources in each cell due to the small size. Each of the sources will emit n_i loops until the back stress (τ_b) of the pile up on the source equilibrates the applied stress ($\tau_i - \tau_1$).

When there are N source, the average strain ϵ is found to be [12]

$$\epsilon = \frac{2Nd^3}{CG\tau_1} (\tau - \tau_1)^2. \quad (3)$$

Using the relation for τ_1 [equation (2)] the stress in the cell can be written for small d as

$$\tau_{\text{cell}} = \tau_0' + \frac{CG}{d^2} \sqrt{\frac{\epsilon b \ln\left(\frac{d}{2b}\right)}{2\pi N}}. \quad (4)$$

If all the cells are deforming at higher degrees of deformation the pile-up will exert such a force at the head that in the next cell new dislocations are generated. This leads to the well-known Hall-Petch relation.

The walls will also bear some stress, which can be accounted for by the rule of mixtures

$$\tau = f_{\text{wall}} \tau_{\text{wall}} + f_{\text{cell}} \tau_{\text{cell}} \quad (5)$$

where f_{wall} and f_{cell} denote the area fractions of walls and cells, respectively, in the glide plane of dislocations.

From the slope of these curves (Fig. 8) it is possible to calculate the density of sources N , which are found to be $3/\mu\text{m}^3$ and $2/\mu\text{m}^3$ in Al-4Si and Al-7.5Si, respectively. Taking for the strain a value of 0.152, the predicted number of dislocation sources lies in the order of $10/\mu\text{m}^3$, i.e. a few dislocations/cell, which indicates that the pile up model describes the deformation process satisfactory within the measuring accuracy. Although residual stresses have some influence on the hardness, this is a small effect compared to the errors in the measurements. Due to the rapid resolidification it is possible that a higher solid solubility is found, however no large variations within the scan velocity range were detected. For larger degrees of deformation, i.e. higher stresses the walls will start to deform in which case the pile up description is not valid any more.

It should be emphasized that the absolute values of the dislocation densities observed at the surface and in thin foils are always questionable in particular in pure Al. However, it is reasonable to assume that the observations still reflect the trends and differences between the various materials studied.

5. CONCLUSIONS

During the cooling period in a laser treatment deformation of the material occurs, together with annealing of dislocations. In pure Al these annealing processes are fast enough to avoid large differences in dislocation density. In Al-Si alloys the dislocation density rises with higher scan velocities probably caused by the smaller distances between the eutectic cells walls. In these alloys entangled dislocation structures were not found in contrast to pure Al.

In pure Al a large number of dislocation loops were found especially at higher scan velocities. During annealing only at laser scan velocities above 2 cm/s a large quantity of dislocation loops became visible.

Both results indicate that at high laser velocities vacancies are frozen in, but at laser velocities around 1 cm/s there is still enough time at high temperature to reduce the vacancy concentration towards lower super-saturation.

For solidification structures consisting of an eutectic material with a high hardness as for an eutectic structure in Al-Si alloys the hardness can be described by a pile up mechanism, which depends on the difficulty to exert stresses on neighbouring cells due to thick and hard walls. The hardness can be described by a $1/d^2$ dependence, i.e. it is mainly determined by the small size of the solidification structure.

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