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Praktischen Metallographie

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Herausgegeben von G. Petzow

Fortschritte in der Metallographie

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geeigenschaften im 3-dimensionalen Raum. Da die Analyseroutinen automatisiert sind, ist es möglich, ganze REM-Bilderserien schnell und mit sehr geringem Arbeitsaufwand quantitativ auszuwerten und somit trotz hoher Vergrößerungen eine statistisch aussagekräftige Fläche zu analysieren. Des Weiteren ist eine Übertragung der Routine auf Bilder ähnlicher Mikrostrukturen möglich. Hierbei können eventuelle Parameteranpassungen in einzelnen Schritten notwendig sein, jedoch ist der Aufwand dafür relativ gering. Der erzielbare hohe Bilddatendurchsatz ist ein enormer Vorteil der vorgestellten Methodik gegenüber anderen hochauflösenden Untersuchungsmethoden, wie zum Beispiel Electron Backscatter Diffraction (EBSD) Messungen. EBSD ist ein exzellentes Tool für die qualitative Charakterisierung des Gefüges, aber der entscheidende Nachteil ist der benötigte hohe Zeitaufwand für die Aufnahme der Beugungsbilder, durch den die Technik im Vergleich zur Rasterelektronenmikroskopie extrem langsam wird. Dadurch ist die quantitative Analyse einer statistisch relevanten Fläche mittels EBSD meist mit zu hohem Zeitaufwand verbunden und stößt bei Gefügen mit Phasen ähnlicher Kristallstruktur schnell an ihre Grenzen.[Day00]/[Hum99] Ein weiterer Vorteil automatisierter Analyseroutinen ist ihre Unabhängigkeit von Umgebungsparametern. Das benötigte Fachwissen wird während der Programmierung und der Erstellung der zu detektierenden Klassen eingebracht und während der Analyse nicht mehr modifiziert.[Ger08] Damit wird die Analyse unabhängig von äußeren Faktoren wie den Lichtverhältnissen, der Wiedergabequalität des Monitors oder dem unterschiedlichen Wissensstand verschiedener Anwender.

Fazit

Ein neues, objektbasiertes Verfahren zur quantitativen Analyse komplexer Mikrostrukturen wurde vorgestellt und auf REM-Bilder verschiedener Mehrphasengefüge angewendet. Mit der vorgestellten Methode ist es möglich, Daten über Morphologie, Kontext und Textur der vorhandenen Bildelemente quantitativ zu erfassen. Durch die Automatisierung und Übertragbarkeit der Analyseroutinen ist eine sehr schnelle und von äußeren Faktoren unabhängige Analyse großer Bildermengen möglich. Damit können auch bei hohen Vergrößerungen statistisch relevante Flächen analytisch erfasst werden.

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Characterization of the microstructure evolution of near beta titanium alloys during hot deformation

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Abstract

Near β titanium alloys seem to be on a gradual increase for structural applications in the aerospace industry because they exhibit relatively good workability due to the low beta transus temperature, deep hardenability and good corrosion resistance. They reach strength values close to those of steel depending on the microstructure obtained during the thermomechanical process. In this work a quantitative analysis of the microstructural changes in two near β alloys (Ti-5Al-5Mo-5V-3Cr-1Zr and Ti-10V-2Fe-3Al) during hot deformation in the α+β field near to the transus temperature is presented. Microstructural features such as shape and maximum lengths of α grains are measured by SEM/BSE from specimens with different grades of deformation at strain rates of 0.01 and $1s^{-1}$. The α grains orient more perpendicular to the compression load with increasing deformation. A fragmentation of the α grain chains followed of a globularization is observed at $0.01s^{-1}$ in Ti-10V-2Fe-3Al. Refinement of β subgrains is observed at high strain rates. Formation of new β grains are determined by EBSD. The changes of the microstructure of these two-phase materials are presented as function of temperature, deformation degree and rate.

1. Introduction

The 15% of the components of the new generation of commercial aeroplanes will be made of titanium. These will be mainly structural parts, such as landing gears and wing structures [1,2]. The use of near beta titanium alloys for this application is growing due to the exceptional strength to weight ratio, corrosion resistance and deep hardenability [3]. The low beta transus temperature and the workability of these alloys make them appropriate for forging. Careful process control and profound knowledge of the influence of processing parameters on the microstructure are of significant importance for the properties of those titanium alloys. Previous works with near beta titanium alloys Ti-10V-2Fe-3Al (Ti-10-2-3) and Ti-5Al-5Mo-5V-3Cr-1Zr (Ti-5-5-5-3-1) [4-6] showed the sensitivity of the microstructure to temperature, time and deformation. This work analyses the microstructure of these alloys quantitatively during deformation in α+β field near to the transus temperature and its relationship with the processing parameters described by the Zenner-Hollomon parameter.

2. Experimental

The near beta titanium alloys Ti-10-2-3 and Ti-5-5-5-3-1 were studied. Table 1 shows the chemical composition of these alloys in weight percent as well as the temperature of phase transformation (T_{β}) [7,8]. Both as received titanium alloys were double melted and subsequently open die forged (cogged) in the α+β and β field with a final step in the α+β field, and annealed before delivery.

Table 1 Chemical composition of the Ti alloys used (wt. %) and temperature of phase transformation (T_β) [7,8]

Alloy	Al	V	Fe	Mo	Cr	Zr	C	O	N	H	T_β [°C]
Ti-10-2-3	3.25	9.24	1.86	-	-	-	0.023	0.12	0.011	0.0008	808
Ti-5-5-5-3-1	5.51	5.04	0.32	5.01	2.85	1.13	0.005	0.066	0.009	0.001	803

Cylindrical specimens were deformed at 0.01 and 1 s^{-1} of strain rate and at 0.1, 0.2 and 0.7 of global true strain by means of a Gleeble® 1500 machine in argon atmosphere at temperatures 20 and 40°C below T_β and water quenched in situ to freeze the microstructure [4-6]. Specimens were cut parallel to the load axis, mechanically ground and polished as described in Table 2. This OPS for titanium alloys consists of 96 parts of distilled water, 2 parts of hydrogen peroxide and 2 parts of ammonia solution (NH3). The microstructure was examined in a Scanning Electron Microscope (SEM) in the Backscattered Electron mode (BSE) to distinguish the α from the β phase. The magnification of the analyzed pictures was 1000X and 2000X with the purpose to reduce the error of measurement during quantitative analysis. SEM images were considered at different positions from the border to the centre along the central diameter for each deformed specimen. The analysis of the α grains was carried out using the software Axio Vs40 v4.4, obtaining area, feret maximum length (FMax), aspect ratio (FMax/FMin) and angle of the maximum length of each grain. The β subgrain size was calculated using the mean linear intercept method according to the ASTM Standard [9]. A mean β subgrain diameter was determined in both perpendicular and parallel directions to the compression axis. Electron Backscatter Diffraction (EBSD) was used to determine the changes in the β phase, observing the β sub-grains after large deformations (0.7 of global true strain).

Table 2. Description of the steps for automatic grinding and polishing

Steps	Consumables	Time [s]	Force [N]
1.Grinding	220 Piano*	Up to plain surface	135
2.Fine Grinding	MD-Largo* (9μm)	600	170
3.Polishing	OPS	600	65

* trademark of Struers

The microstructural features (size, orientation) were correlated with the local effective strain and strain rate obtained by simulation, using the commercial finite element code DEFORM™ 2D. The deformation produces barrelling of the compression samples provoked by the axial gradient of temperature and by friction with the anvils. Values up to 1.4 of effective strain and 3.7s^{-1} were reached at the centre of the sample.

3 Results

3.1. Alpha grains behaviour

The dissolution of the acicular α phase into β phase during heating of an initial bimodal condition in Ti-10-2-3 as well as a globular microstructure in Ti-5-5-5-3-1 was reported in previous works [4-6]. The stabilization of the α content was achieved after 300s. After deformation of Ti-10-2-3 at lower effective strain rates, a slight increase of the mean value of the aspect ratios of α grains at lower local deformation (<0.5) was determined as illustrated Figure 1a) (bars represent the standard deviation of the distribution). This can be related to the fragmentation and subsequent deformation of the chains of α grains indicated by an increment of the number density of α grains (Figure 1b)), especially at lower temperatures (768°C). At strains higher than 0.8, the number density of grains

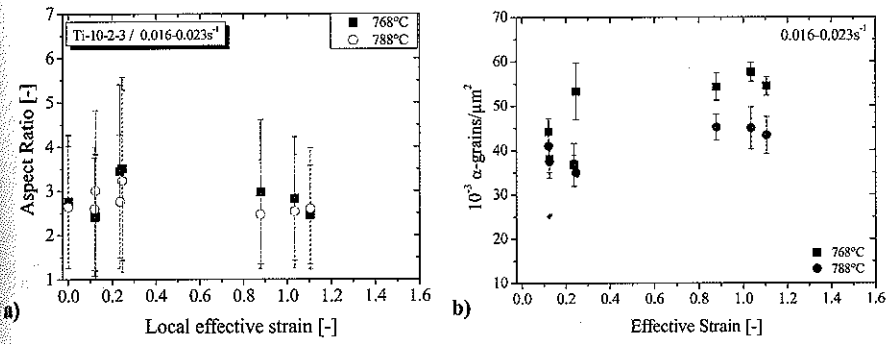

Figure 1. Alpha grains behaviour with effective strain in Ti-10-2-3 during deformation at 768°C and 788°C at global strain rate of 0.01s^{-1}: a) mean value of the aspect ratio distribution b) number density of alpha grains.

was almost constant but a decrease of the aspect ratio denoted globularization of α grains controlled by diffusion at 768°C.

Elongation of α grains was observed at high effective strain rates (>1s^{-1}) in Ti-10-2-3 and in Ti-5-5-5-3-1 for the whole range of temperatures and strain rates. Figure 2 shows examples of both alloys, where the mean value of the aspect ratio distribution increases with increasing effective strains.

The orientation of the α grains changed during deformation (Figure 3); they aligned preferentially perpendicular to the load. When the local strain increased, the orientation of the grains increased concentrating the majority of the grains within a range of angles of ±60° for effective strain rates below 0.023s^{-1} and ±70° for those compressed at more than 0.8s^{-1} with respect to the load.

The Zenner-Hollomon parameter, defined as $Z=\dot{\varepsilon}\, e(-Q/RT)$, was related to the microstructural features and shown in Figure 4. For both materials there are no significant changes in the α grains morphology and size for the range of strain rate and temperatures studied (at 0.63 of local deformation, for example). A slight increment in the aspect ratio (A) in Ti-10-2-3 is observed as a consequence of the elongation of α grains by increasing Z.

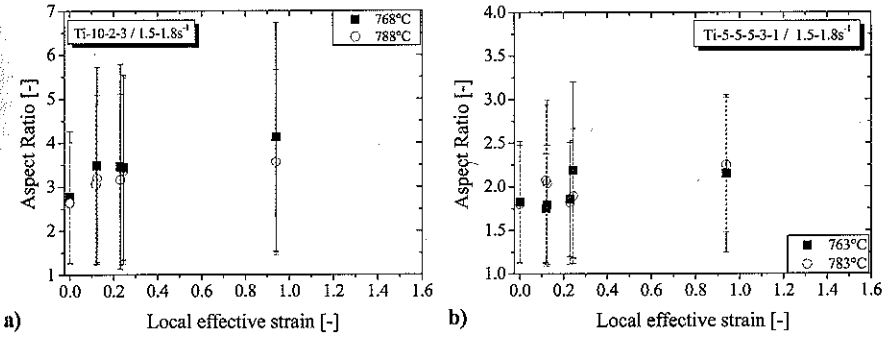

Figure 2. Mean value of the aspect ratio distribution during deformation at global strain rate of 1s^{-1} in subtransus temperature for: a) Ti-10-2-3 and b) Ti-5-5-5-3-1.

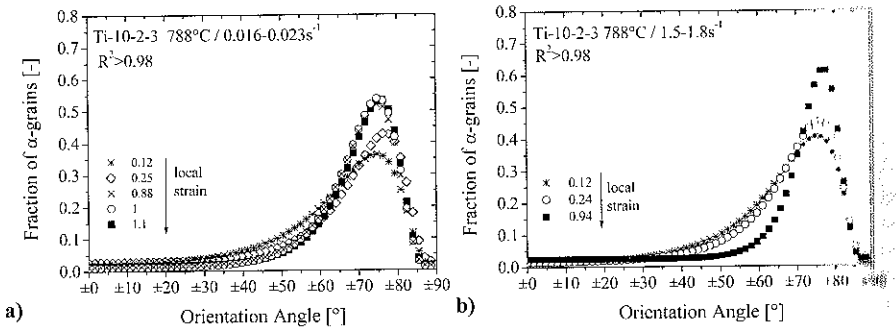

Figure 3. Example of the orientation angle distribution of the maximum length of the alpha grains in Ti-10-2-3 with respect to the applied load for different local deformations at 788°C: a) 0.016-0.023s^{-1}, b) 1.5-1.8s^{-1}.

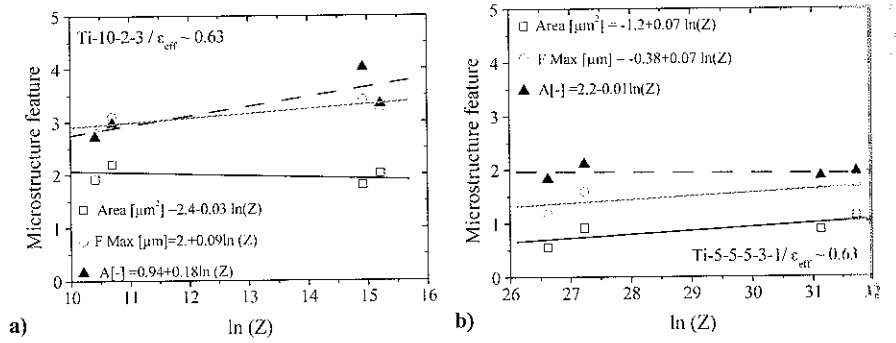

Figure 4. Relationship between Z parameter and α-grains microstructural features (size, maximum length, aspect ratio) at 0.63 of effective strain of: a) Ti-10-2-3 and b) Ti-5-5-5-3-1.

3.2. Beta subgrains and grains behaviour

The microstructural behaviour of the β phase by deformation can be summarized in Figure 5 and Figure 6. Compared to the initial state (Figure 5a)), subgrain size becomes bigger after deformations at 0.01s^{-1} and smaller at 1s^{-1}. For both global conditions, the subgrain size seems to be independent of the strain if larger than 0.6. This invariance in the size is related to the steady state condition. The subgrains varied strongly with the Z parameter, decreasing in size with Z increases, as illustrated in Figure 5b). Compared with α phase, it denotes that most of the deformation occurred in β phase. Figure 6 compares the β grains (with different grey scale) before and after deformation at 0.01s^{-1} by EBSD measurements. The formation of new grains was observed during deformation. This phenomena is related to the progressive increase in the misorientation of the β subgrains produced by dynamic recovery and the effect of the second phase (α) as a harder particle dispersed in the matrix increasing the formation of high angle boundaries [10] and strain concentration in the beta phase. This mechanism is called continuous recrystallization and occurs at larger deformations.

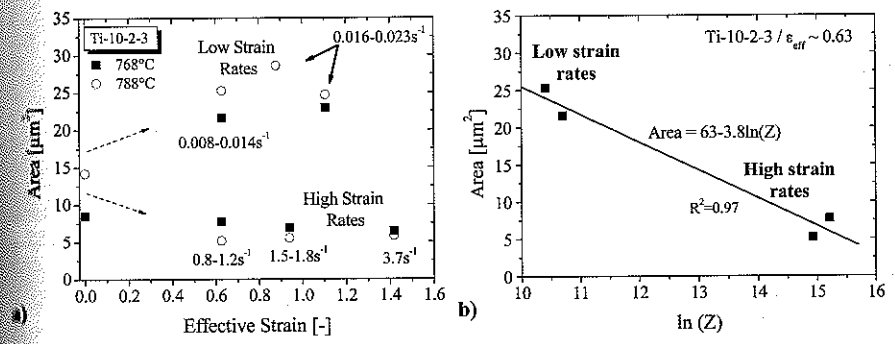

Figure 5. Example of beta sub grain size in Ti-10-2-3 with: a) effective strain for different effective strain rates and b) with Z parameter at 0.63 of effective strain

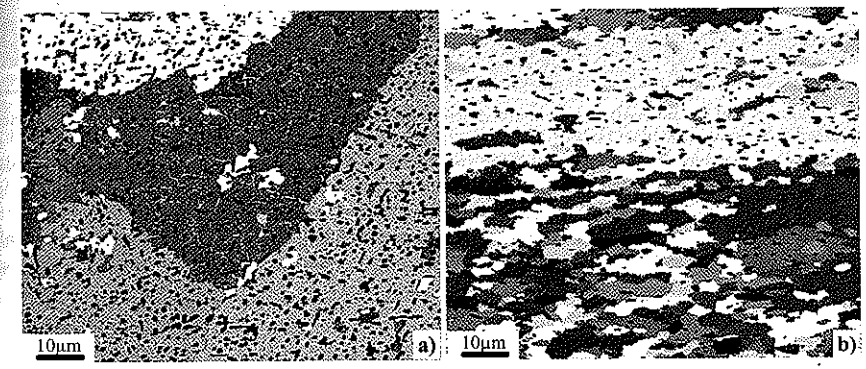

Figure 6. Beta grains observation by EBSD in Ti-5-5-5-3-1 at 763°C: a) without deformation and b) deformed at 0.01s^{-1} and 0.7 of true strain. Compression axis is vertical. α grains are black.

Figure 7 shows an overview of the different microstructures at the centre of the specimens before and after deformation in both alloys at 0.7 of global true strain and different strain rates. α grains (dark) are elongated in Ti-5-5-5-3-1 after deformation (b and c) in comparison with the not deformed state (a). At low strain rate in Ti-10-2-3 (e), the α grains become more globular compared to the initial state (d) while the alpha phase is squeezed and elongated at 1s^{-1} (f). The orientation, almost perpendicular to the load, of α-grains as well as the changes in size of the β subgrains (bright) can be observed.

4. Conclusions

The analysis of SEM images of hot compressed samples allowed the quantification of the microstructural features of α-grains (size, orientation evolution, etc) and the β subgrains size evolution. Using Electron Backscatter Diffraction (EBSD) showed that this technique is appropriate for the identification of β grains.

α grains in Ti-5-5-5-3-1 become elongated with increasing strain, while the chains of α grains were fragmented followed by a globularization by diffusion at 0.01s^{-1} in Ti-10-2-3. An effect of flattening of the α grains at 1s^{-1} global strain rate was observed.

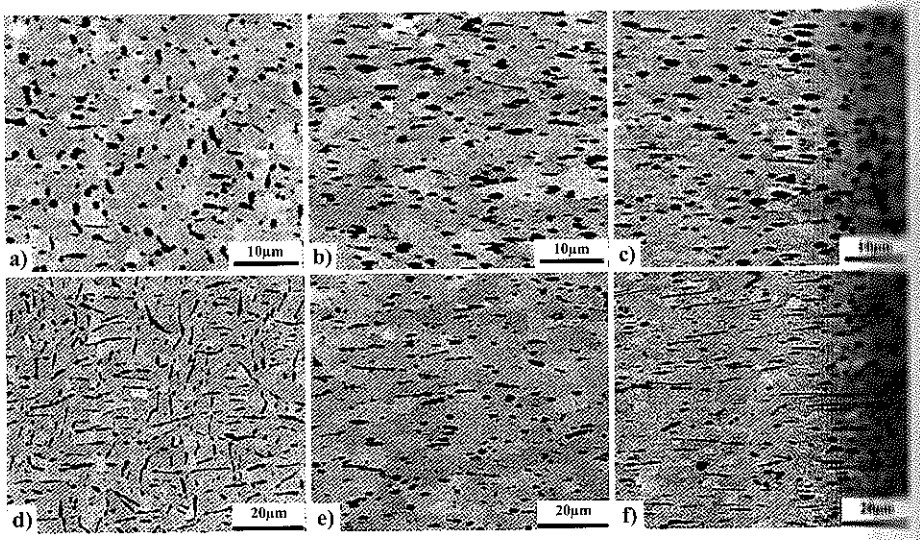

Figure 7. Summary of the microstructure obtained before and after deformation at the centre of the specimen. Compression axis is vertical. For Ti-5-5-5-3-1 at 763°C: a) without deformation, b) and c) deformed to 0.7 at 0.01s^{-1} and 1s^{-1}, respectively. For Ti-10-2-3 at 768°C: d) without deformation, e) and f) deformed to 0.7 at 0.01s^{-1} and 1s^{-1}, respectively.

β subgrains decrease strongly when Z increases, indicating that the deformation occurred mainly in the β phase for this range of temperature. Formation of new grains at high deformations suggests continuous recrystallization.

5. Acknowledgments

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Grundlagen der Reinheitsgradbestimmung nach der neuen Norm DIN EN 10247:2007 „Metallographische Prüfung des Gehaltes nichtmetallischer Einschlüsse in Stählen mit Bildreihen".

Damian Moll, AG der Dillinger Hüttenwerke, Dillingen / Saar

1 Entstehung und Entwicklung der neuen europäischen Norm

Im Zusammenhang mit der europäischen Normung der Anforderungen an Stähle entstand der dringende Bedarf, ein einheitliches Verfahren zur Bestimmung des Gehalts an nichtmetallischen Einschlüssen festzulegen. Deshalb wurde im November 1988 auf einer Sitzung von ECISS/TC 1A beschlossen, eine Arbeitsgruppe „Mikroskopische Prüfung von Stählen auf nichtmetallische Einschlüsse" zu gründen. Frankreich, Schweden, Finnland, Großbritannien und Deutschland hatten Experten als Mitarbeiter für die Arbeitsgruppe benannt. Diese Arbeitsgruppe hat im Rahmen von acht Sitzungen einen Europäischen Vornorm-Entwurf prEN V 10247 „Metallographische Prüfung des Gehaltes nichtmetallischer Einschlüsse in Stählen mit Bildreihen" erarbeitet, der Arbeitsweisen für die mikroskopische Kennzeichnung von nichtmetallischen Einschlüssen im Stahl unter Verwendung von Richtreihenbildern festlegt. Diese Vornorm basiert auf der Prüfmethode der bisher angewandten nationalen Normen. Der Vornorm-Entwurf wurde im Juni 1998 als Vornorm DIN ENV 10247 veröffentlicht. Ab diesem Zeitpunkt konnten Anwender Erfahrungen mit dem neuen Verfahren sammeln und sie den entsprechenden nationalen Normungsstellen mitteilen. Als Beispiel kann der Erfahrungsaustausch, der im Rahmen einer Arbeitsgruppe ENV 10247 des VDEh Unterausschusses für Metallographie, Werkstoffanalytik und –simulation stattgefunden hat, genannt werden. Die aus dieser Zusammenarbeit gewonnen Erkenntnisse wurden als Grundlage für die deutsche Stellungnahme verwendet. Die vorgeschlagenen Änderungen der Norm wurden im Herbst 2004 durch Experten der nationalen Delegationen diskutiert und akzeptiert, so dass damit die endgültige Abstimmung der Norm angegangen werden konnte.

Die neue europäische Reinheitsgradnorm wurde als gültige Norm DIN EN 10247 „Metallographische Prüfung des Gehaltes nichtmetallischer Einschlüsse in Stählen mit Bildreihen" im Juli 2007 veröffentlicht.

Wegen der Umstellung in der Betrachtungsweise der Einschlüsse sowie wegen der notwendigen (gerätespezifischen) Umrüstungen der Mikroskope und zur Entwicklung einer entsprechenden Software für Bildanalysesysteme wurde eine Übergangszeit von 2 Jahren zur Einführung der neuen und bis zum Zurückziehen der alten Norm DIN 50602 beschlossen.