BRITISH STANDARD

BS EN 3114-001:2006

Aerospace series — Test method — Microstructure of $(\alpha + \beta)$ titanium alloy wrought products —

Part 001: General requirements

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ICS 49.025.10

British Standards

National foreword

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A list of organizations represented on ACE/61/-/49 can be obtained on request to its secretary.

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Contents

Foreword

This document (EN 3114-001:2006) has been prepared by the Aerospace and Defence Industries Association of Europe - Standardization (ASD-STAN).

After enquiries and votes carried out in accordance with the rules of this Association, this Standard has received the approval of the National Associations and the Official Services of the member countries of ASD, prior to its presentation to CEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2007, and conflicting national standards shall be withdrawn at the latest by June 2007.

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Introduction

This standard is part of the series of EN metallic material standards for aerospace applications. The general organization of this series is described in EN 4258.

1 Scope

This standard specifies the conditions for micrographic examination of ($\alpha + \beta$) titanium alloy wrought products and description of terms used.

Specific microstructures applicable to each type of product are defined in EN 3114-002 to EN 3114-004.

This standard shall be applied in conjunction with EN material standards, which define the acceptance criteria unless otherwise specified on the order.

It is applicable to:

- bars, sections, forging stock and forgings (EN 3114-002);
- plate (EN 3114-003);
- sheet for superplastic forming (EN 3114-004).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 3114-002, *Aerospace series — Microstructure of* ($\alpha + \beta$) *titanium alloy wrought products — Part 002: Microstructure of bars, sections, forging stock and forgings.*

EN 3114-003, *Aerospace series — Microstructure of* ($\alpha + \beta$) *titanium alloy wrought products — Part 003: Microstructure of plate.*

EN 3114-004, *Aerospace series — Microstructure of* ($\alpha + \beta$) *titanium alloy wrought products — Part 004: Microstructure of sheet for superplastic forming.*

EN 4258, *Aerospace series — Metallic materials — General organization of standardization — Links between types of EN standards and their use.*

3 Sampling

The sampling and its frequency shall be as defined in the relevant technical specification and/or order.

The sampling sections shall be at least 10 mm, in thickness or length.

Samples shall preferably be taken by sawing. Cold work hardened zones shall be avoided or removed. If the sampling material is taken by abrasive cut off or flame cutting, the resulting heat affected zone shall be completely removed in the course of sample preparation.

4 Preparation of the microsections

4.1 General

The surface of the sample sections to be examined shall be prepared by machining and grinding and polishing to ensure that the microstructure is clearly defined after etching.

In the case of electrolytic polishing, grinding may be omitted.

The method of preparation shall not change, contaminate or deform the surface microstructure.

4.2 Grinding

Resin bonded alumina, zirconia or silicon carbide paper shall be used for grinding. The pressure shall be as low as possible in order to avoid any cold work hardening of the surface of the microsection.

4.3 Polishing

Polishing may be accomplished mechanically and/or electrolytically.

The following solution is recommended for electrolytic polishing:

- 700 ml methanol;
- 200 ml butylglycol;
- 60 ml perchloric acid.

4.4 Etching

The etchants and their typical composition indicated in Table 1 may be used for development of microstructure.

Table 1 — Etching of titanium alloys

	Reagent	Vol. %	Remarks	Use
1	Hydrofluoric acid (40 %) Nitric acid (65 %) Water up to	8 40 100	Swab or immerse	Micro etch Macro etch (Etching time up to several minutes)
2	Hydrofluoric acid (40 %) Nitric acid (65 %) Water up to	2 10 100	Swab or immerse	Bright grain boundary etch
3	Hydrofluoric acid (40 %) Benzalkonium chloride Methyl alcohol Glycerol	2 to 6 19 35 40	Swab Avoid overetching Avoid water in etch solution	Stain etch Stain α or β depending on the heat treatment condition
4	Hydrofluoric acid (40 %) Water up to	0,5 100	Swab or immerse	Stain etch
5	Hydrofluoric acid (40 %) Nitric acid (65 %) Glycerol up to	2 to 20 5 to 25 100	Swab Glycerol acts as inhibitor	Bright etch Slower etch than 1
6	Potassium hydroxide Hydrogen peroxide (30 %) Water up to	10 15 100	Immerse (70-80) °C	Grain boundaries, subgrain boundaries. Stains α and transformed β, retained β not stained. Orientation sensitive, develops contrast between grains.
7	Hydrofluoric acid (40 %) Nitric acid (65 %) Lactic acid	20 20 60	Swab rigorously	Chemical polish and etch
8	Hydrochloric acid (40 %) Nitric acid (65 %) Lactic acid	10 25 65	Swab	General etch
9	Hydrofluoric acid (40 %) Nitric acid (65 %) Water up to	10 15 100	Swab or immersion + 1 min in ammonium bifluoride 20 g/l	Etch for segregation
10	Nitric acid (65 %) Water up to	25 100	Swab or immersion	Removal of copper surface layer
11	Hydrofluoric acid (40 %) Nitric acid (65 %) Hydrogen peroxide (30 %) Water up to	1 2 50 100	Spray wash	Removal of tin surface layer

5 Assessment

The prepared surface shall be examined at the same magnification as that of the reference photomicrograph.

6 Description of microstructures

6.1 Acicular α

A transformation product arising, during cooling, by nucleation and growth on preferred planes of the primary β phase, or by martensitic transformation and growth. It appears in acicular form, as pointed lamellae, or plates.

6.2 α enriched zone (Blocky α)

A coarse area of α by comparison with the primary α grain of the matrix. Such α areas are frequently accompanied by grain boundary α. Microhardness is not significantly different from that of the matrix.

6.3 α phase (α)

The stable sub-β transus α phase with close packed hexagonal crystal structure.

6.4 α + β structure

Microstructure containing α and β phases in various forms.

6.5 α stabilized hard zone High Interstitial Defect (HID)

Zone of higher α stabilized content, significantly harder than the surrounding structure. Normally caused through local oxygen and/or nitrogen enrichment.

6.6 α stabilized surface (α case)

A surface zone enriched mainly by oxygen or less often by nitrogen or carbon, in which the α phase is stabilized. The α case result from elevated temperature exposure to environments containing these elements. α case is hard, brittle and considered detrimental.

6.7 α stabilizer

Element preferentially dissolving in the α phase, and which increases the α and β transus temperatures.

6.8 α stringer

In comparison with the fine α + β matrix, clearly defined coarse elongated α phase.

6.9 α transus

The temperature which determines the boundary between α and α + β fields - also known as α/α + β transition temperature.

6.10 Aluminium enriched zone

Zones in which an increased amount of α-stabilizing aluminium leads to higher percentage of α-phase compared with the matrix.

In comparison with the matrix structure, such areas show relatively little hardness increase, also called soft α.

6.11 Banded structure

Structure in which the components are orientated in the direction of maximum work in a fibrous plate-like form.

6.12 Basket weave

α plates or lamellae with or without β phase between them. In this case the α phase is oriented in "groups" like a woven basket (Figure 1).

6.13 β eutectoid stabilizer

Alloying element which dissolves preferentially in the β phase and lowers the β transus temperature. The β phase may decompose via an eutectoid reaction, into α + intermetallic compound.

6.14 β fleck

Zones of low or zero primary α in an α + β structure. They have a noticeably lower β transus temperature than surrounding structure.

6.15 β grain size

After transformation from the β field, β grain size can be retained as the macro grain size. With α + β working, the shape of the original β grains may be changed.

6.16 β soluble isomorphous element

An element showing continuous solubility in the β phase.

The β transus temperature is lowered, without an eutectoid reaction.

6.17 β phase (β)

The stable phase above the β transus, having body centered cubic crystal structure.

6.18 β stabilizer

Element dissolving preferentially in the β phase, and which lowers the β transus temperature.

6.19 β transus

The temperature which specifies the boundary between the α + β range and the β range, also designated as the α + β/β transformation temperature.

6.20 Colonies

Zones within prior β grain boundaries, and having parallel α plates.

6.21 Elongated α

α phase in fibrous form, arising primarily from unidirectional deformation.

6.22 Equiaxed α

α with a rounded or polygonal appearance with similar dimensions in all directions.

6.23 Globular α

α phase, primarily showing circular shape.

6.24 Grain boundary α

The α phase appearing at prior β grain boundaries. It can encompass the complete prior β grain.

6.25 Hydride phase

Brittle intermetallic compound of composition TiH_X. It can appear when the solubility limit for hydrogen is exceeded.

6.26 Intermetallic compound

A compound formed between alloying elements in an alloy system with at least one field of limited solubility.

6.27 Interstitial element

Element of relatively small atomic diameter (e.g. oxygen, nitrogen, carbon, hydrogen) which can occupy sites between the titanium lattice.

6.28 Lamellar α

Parallel orientation of elongated phase.

6.29 Lean α area

See 6.14 β fleck.

6.30 Martensitic α

A supersaturated α phase, not in equilibrium, arising from diffusionless transformation of the β phase (Hexagonal α or orthorhombic α).

It is often difficult to distinguish martensitic α from acicular α.

6.31 Matrix

Matrix is the uniformly distributed microstructural background. The matrix can be single or multiphase.

6.32 Omega phase (ω)

Omega is an intermediate phase which can arise from transformation of metastable β into equilibrium α. It cannot be seen under an optical microscope. In alloys with β content or in metastable β alloys it can occur at ageing temperatures and lead to a decrease in formability.

6.33 Primary α

α phase arising through recrystallisation or growth following hot forming or annealing in the α + β field.

6.34 Plate-like α

Coarse α phase which precipitates on preferred planes of the β phase during transformation from β to α + β, frequently in colonies or a Widmannstätten structure.

6.35 "Serrated" grains

Irregular α grains with grain boundaries of a sawtooth nature.

6.36 Substitution element

An element which by virtue of a similar atomic diameter similar to titanium, can replace titanium atoms in the lattice.

6.37 Transformed structure

This refers to α + β structures produced by nucleation and growth via transformation from the β phase. Transformation occurs during cooling from temperature above the β transus. The transformed structure consists of α platelets or in multiphased structures, of α platelets with β phase between them.

6.38 Widmannstätten structure

A structure of α - or α and β platelets in a crystallographically orientated geometrical arrangement, arising under particular conditions during cooling from temperatures above the β transus (see Figure 2).

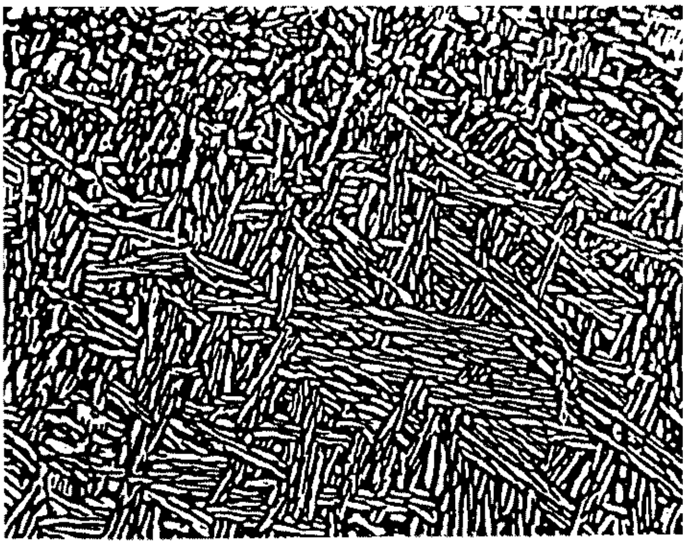

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Figure 1 — Basket weave

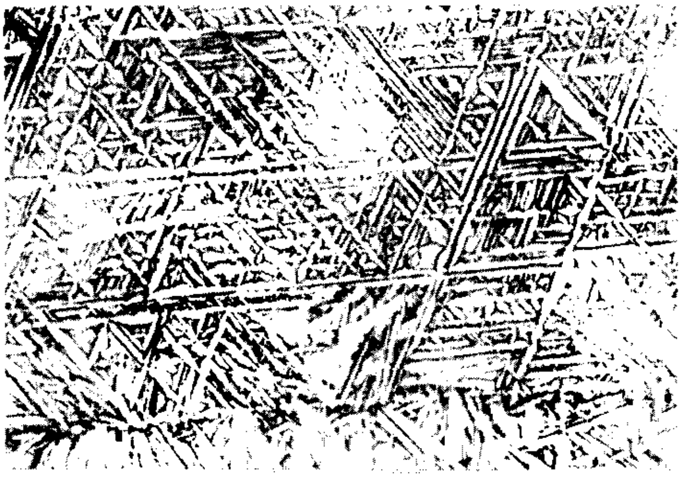

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Figure 2 — Widmannstätten structure

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