TITANIUM'99: SCIENCE AND TECHNOLOGY

Electron Beam Cold Hearth Melt of Titanium and its Alloys

V.FEDOROV, N.LASHUK, V.SCHEKIN-KROTOV Joint Stock Company "FIKO", Kyiv, Ukraine

V.TREFILOV, A. MINAKOVA, N.MINAKOV, V.MINAKOV Institute for Problem of Material Science, National Academy of Science, Kyiv, Ukraine

ANNOTATION

JSC "FIKO" has developed one fold electron-beam cold hearts process. It can be used for manufacturing of ingots $\emptyset 400$ mm and 630mm, 2 m long, up to 2,5 tons by weight. Two 180 KW cold cathode glow discharge electron guns are used for charge remelting and two 90 KW guns are applied for maintenance of necessary melt temperature. The process of melting goes in electron-beam cold hearts furnace through intermediate poll, charge in the container (in large scale scraped condition) moves horizontally. This method allows to use up to 100 % scrap and wastes of titanium industry as raw material.

The chemical content of alloys is true to the specified for BT1.0 (Grade 2) and BT6 (Grade 5) analyses, and uniform by the length and diameter of ingot. The process can be used to refine titanium from nitrogen and embedded impurities and to remelt low-grade sponge and the wastes of titanium industry. The properties of alloys BT1.0 (Grade 2) and BT6 (Grade 5) satisfy to the requirements of the standards. There were no impurities that exceeds critical size (5-20 microns) found on the fractured surfaces of alloys investigated. The specimens has been broken in the Auger spectrometer column under high vacuum condition. Auger spectra does not indicate presence of interstitial impurities Na, K, Ca, Cl, P, S, which are present in initial charge materials and in vacuum arc remelting metal. Such impurities are not regulated by the standards. These elements are capable of forming partition borders, decreasing the effective fracture energy and in some cases are responsible for plasticity and embrittlement.

Among conventional processes of titanium ingot production, performance specification of Vacuum Arc Melting (VAR) is the best, the one of Electron Beam (EBM) process is ranked after (1). Development of melt processes with application of cold hearth and independent heat sources was encouraged by complexity of consumable electrode manufacturing and necessity of double -or triple remelting process to guarantee high quality of the ingot. A lot of investigations were implemented by Ukrainian scientists (Paton Electric Welding Institute, Ukraine) to improve electron beam melt processes for titanium ingot production (2, 3, 4).

The author (5) emphasizes the high refining ability of EBM of titanium and its alloys. It was shown that nitrogen content falls to 50 ppm and chlorine one - to 8 ppm. In the subsequent studies special attention was paid to application of cold hearts with ingot withdrawing at the bottom that follows.

The trend of the titanium metallurgy development is direct melting of titanium sponge, scrap and waste using technology based on cold hearth process (sometimes it is called as a cut-in vessel). The leading companies use this approach. Electrode preparation is not required for the charge melt by the process in question. Electron beam and plasma guns, inductive heating in subdivided water-cooled crucibles are involved to the process as independent sources of heat

Application of open molt pool allows the metal to be refined effectively. The results of cold hearth process application for titanium ingots production are presented in $(^6, ^7, ^8, ^9, ^{10})$. The cold hearth melt process eliminates hard α -phase and hard density inclusion formation when compared to the triple VAR. But the mechanism of chemical segregation formation has not been revealed yet.

In it necessary to pay special attention to the ways of plasticity enhance due to targeted control of:

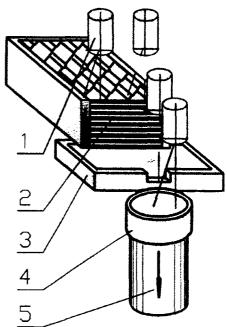
- grain boundaries spots formed by insoluble elements in the solid state;
- inclusions of various size:
- segregations at the interfaces in polycrystalline alloys. In this case we mean segregation of impurities such us Na, K, Ca, S, Cl but not main alloying elements.

Profound investigation on failure reasons of aerospace hardware had been implemented by a group of scientists at the IPMS, Ukraine during nearly 30 years. One of the found causes is the presence of Na, K, Ca, S, Cl at the initial β -grain boundaries as well as nitrogen and oxygen (11,12,13,14,15),. These elements moderate the cohesive strength along the boundaries, that leads to plasticity loss and alloy embritlement. According to Auger spectroscopy of the fracture surface, content of the above mentioned elements can reach 50% on the surface layers. Metal refining and reduction of the grain size are highly effective means to eliminate harmful effect of the above impurities (Na, K, Ca, S, Cl). Content of these elements is not regulated by the quality standards parameter.

Independent heat sources of Electron Beam and Plasma Melt processes allow to monitor and control the temperature of the molted metal zone and crystallization, to effect the structure and impurities distribution.

Characteristics of Ukrainian raw materials stock are in presence of big amount of conversion titanium scrap and titanium sponge. Because of complexity and high cost of consumable electrode preparation a technology on the base EB process (with independent heat sources) and horizontal charge feed was developed. The technology allows premium quality titanium to be made from titanium scrap and waste, titanium sponge due to elimination of impurities. For example, the titanium made by the technology from the low quality sponge TG-120 has nitrogen content not more than $5x10^{-3}$ wt %.

EBM unit developed by the limited company "FIKO" are presented on figure 1. The technology is based on cold hearth process with horizontal charge feed. Molten metal traverses continuously a horizontal water cooled heart from the feed stock melting region to mold where the ingot solidifies and at the bottom - withdrawn. The main distinctive feature of the technology is application of cold cathode glow discharge electron guns (CDGDEG) instead of common electron beam guns. The four CDGDEGs are used: two of them of 180 kW each are for charge melting, the other two of 90kW each - to maintain the constant melt temperature (fig. 1). Insensibility for the metal sputtering and stable performance at pressure up to 2.5 Pa (16) in the melt chamber are specific characteristic of



Fig, I EBM unit at "FIKO" limited plant. 1 - Cold cathode glow discharge gun; 2 - feed stock; 3 - cold hearth; 4 - mould; 5 - ingot.

the HVGDG. For comparison pressure less than 2.5×10^{-2} Pa is required to guarantee operation of the gun with the thermocathode. Comparative characteristics for different melt processes are listed in table 1.

Table 1. Comparison of different smelting processes of titanium waste recycling

	Parameter	Type of smelting process (17)				Ingot EBM JS "FIKO"	
		VAR	PA	EBCH	PACH	Ø400	Ø 640
l.	Specific electricity consumption, KWh/t	1,100	1,760	3,000	3,000	2,8661	2,0431
2.	Consumed capacity. KVA	2,000	1,200	2,500	2,500	5,00	7,00
3.	Water consumption, litre/min	1,150	1,515	2,275	2,275	1,000	1,000
1.	Water loss litre /t	-	-	-	-	17,000	10,000
5.	Environment protection cost, US\$/kg	<u>-</u>	_	-	-	0.0009	0.0012
 5.	Scrap suitability for the process, %	80	70	50	62	60 70	60 70
7.	Specific scrap content in the consumable electrode, %	45(18)	100(19)	100	100	100	100
3.	Required number of remelting	2	1	1	1	1	1
€.	Output of good product • Single remelting / Double remelting	87.5 /75.7	99.5	93.0	99.0	90.8	89.6
10.	Number of operators	1	2	3-4	3	3	3
11.	Capital cost (VA-1.0)	1.0	1.5	2.5	2.1	2.4	2.5
12.	Cost of production US\$/kg	1.2	1.4	3.3	2.4	1.8	1.35

EBCH and PACH - are electron beam and plasma arc cold hearth remelting.

Consumable electrode cost is omitted due to lack of data. The "FIKO" technology has the following advantages:

- 2-3 times less consumable electric capacity;
- specific electricity consumption is lower that that for EB and VAR processes due to glow discharge gun application;
- lower vacuum level is required for the melt and crystallization processes;
- evaporation of main allowing elements (Al in particular) is reduced;
- control over initial grain size it can be reduced by 2 times;

- higher output of good quality metal in comparison to double VAR if feed stock is 100% titanium scrap;
- the technology is environmentally friendly;
- the technology has all common advantages that proper to the cold hearth process.

A problem on feed stock arises if piece thickness is less that 3 mm and large specific surface takes place (oxidation hazard).

"FIKO" company produces ingots with \emptyset 400 mm and \emptyset 630 mm in diameter, 2 meter large and weight is more than 2.5 t over the one full-scale heat.

Chemical composition of VT1 and VT6 alloys meets requirements of the standards for Grade 2 (VT1) and Grade 5 (VT6) (fig. 2).

It is well known that independent heat sources of EB and Plasma Melts enable to control temperature in melting and crystallization zones, effect the structure and impurities distribution.

Implemented study of "FIKO" made ingot structure has shown that control over the heat parameters in crystallization zone led to reduction of initial grain size at least by two times for the ingots of \varnothing 400 and 600 in diameter. The ingots have equiaxed grains, size of the β -grains is 3-5 mm for the ingot of 400 in diameter (fig.3) and 15-20 mm for 600 mm diameter ingot.

Investigations of VT6 alloy made by the "FIKO" Company were implemented by Auger spectroscopy and REM at the IPMS of NAS, Ukraine. The specimens were prepared for tensile tests: initial β- grain boundary

sited along the fracture zone. Auger specimens were notched along initial β - grain boundaries. Specimens after tensile tests and failure inside of Auger spectroscope column were subjected to fracture character evaluation.. Fig 4 presents the general results for implemented studies. A crack originated from intersection of the initial β-boundary and surface (surface stress concentrator) has a ductile character of propagation along the grain boundary by mechanism of dimple formation and merging. As the crack propagates, it crosses the initial β -grain (fig. 4) and structure consisted of $\alpha+\beta$ grains is

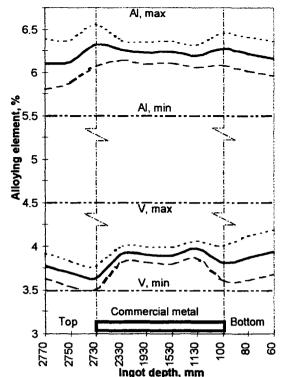


Fig 2. EBM 6-4 alloy ingot composition. The confidence interval is shown schematically with dashed lines. Acceptable interval of homogeneity - with dash-dot lines. Ingots body data are shrinked, top and bottom ends are stretched

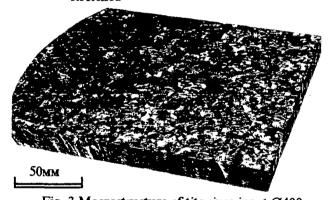


Fig. 3 Macrostructure of titanium ingot \emptyset 400.

observed. The fracture character is ductile. Particles including hard α -phase with size more that critical crack size (1-5 mk) were not detected at the fracture surface.



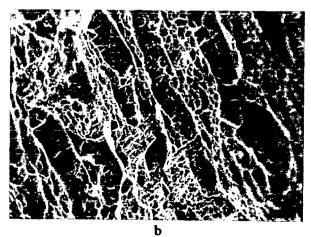


Fig. 4 6-4 alloy fractography: a - fracture source (stress concentrator), ductile fracture through an initial β - grain boundary; b - transcrystalline fracture of an initial grain body, ductile failure of $\alpha+\beta$ phases.

Auger spectrums do not reveal Na, K, Ca, Cl, P, S along the initial grain boundaries. As it was mentioned before, these elements are nod regulated by the standards and presented in feed stock and VAR titanium. These elements are insoluble in titanium and form spots along interfaces that causes specific surface energy fall and consequently plasticity loss together with embrittlement.

Auger analysis of furnace chamber deposit from the area over the cold hearth and ingot mold pool has revealed the above mentioned element presence. These harmful impurities are detected even in the case of VAR titanium scrap and waste remelting. It enables us to state that "FIKO" technology comprising cold hearth process with cold cathode glow discharge electron guns has high refining ability of titanium and its alloys.

Mechanical properties of 6-4 alloy (limited company "FIKO"), processed under the scheme: forging $\varnothing 400 \rightarrow 1000 \div 1050 \circ C \rightarrow \varnothing 250 \rightarrow 1050 \circ C \rightarrow \varnothing 170 \rightarrow 1000 \circ C \rightarrow \varnothing 110 \rightarrow \text{annealing} 800 \circ C t=2 hr, vacuum 6.7x10⁻³ Pa, are listed in table 2.$

Table 2 Mechanical properties of 6 4 alloy.

№	forging cutting out scheme	Strength pi	operties, MPa	%		
<u> </u>	for sample preparation	O0.2	σв	S _f	δ	Ψ
ı	Ø110,	863-877	923-924	1180-1200	14,3 - 15.7	27.1-30,2
2	ASTM B348Grade 5 Ø 76mm	825	895		>10	>25

Properties of the VT-1 alloy (Grade 2) and VT-6 alloy (Grade 5) meet the standard requirements. True fracture stress S_Γ for VT6 alloy made by "FIKO" process and after thermo-mechanical treatment has 1200-1400 Mpa and equals to that for VT6 alloy of VAR process. Parameter S_Γ is sensible for any type of defects responsible for plasticity and strength loss. The parameter can be used as a additional criterion for evaluation of metal quality and technology perfection [20].

CONCLUSIONS

- 1. Unique titanium alloy Hearth Melt "Single-Stage" technology based on cold hearth melt and cold cathode glow discharge electron gun has been developed by "FIKO" company. The technology allows to eliminate segregation of main alloying elements and harmful impurities presence, to reduce the initial grain size: it does not exceed 7x20 mm for \emptyset 400 mm ingot.
- 2. 6-4 titanium alloy ingot of Ø400-600 mm in diameter that meets the standard requirements on heterogenity is produced by "FIKO" single melt process.
- 3. Fracture of the cast 6-4 alloy has ductile character at room temperature, true fracture stress is 140 MPa and equals to one for triple VAR 6-4 alloy.

LITERATURE

- 1. Shigeru Tasamoto. Present features in production of titanium sponge and ingot. Proc. of 7th world titanium conference Titanium92, Science and Technology held in San-Diego, (California, USA). Warrendale: TMS-AIME,.- 1992.- I.- P. 52-63.
- 2. Yu. Kurapov Vacuum refining of metals process through the electron beam remelting. K.: Naukova dumka 1984 168 c. (in Russian)
- 3. Movchan B.A., Tikhonovskiy A.L., Kurapov Yu.A. Electron beam remelting and refining of metals and alloys K.: Naukova dumka 1973 238 p. (in Russian)
- 4. Tikhonovskiy A.L., Tur A.A., Tunik A.V. Electron beam remelting of the titanium sponge is a new titanium ingots and slab process. 1993 (in Russian).
- H.Stephan Schmelz und Reinigungsverfaren im Elektronenstrahl schmelzofen (Teil1) / Metallwissenschhaft und Technic 1975 27 Jahrang Juli Heft 7 P 705-715.
- 6. Bellot J-P, Hans S, Hess E, Edie C, at all The formation and removal of «Hard α» material during the melting of titanium alloys Proc. of 8-th world titanium conference: «Titanium'95,» held in Birmingham (UK), Cambridge: University press. 2.-P. 1454-1461
- 7. Hearth Melt Plus Vacuum Arc Remelt: Production Status W. H. Buttrill, C. E. Shamblen // Proc. of 8-th world titanium conference: «Titanium'95,» held in Birmingham (UK), Cambridge: University press.- 2.-P. 1446

- 8. Shamblen C.E. titanium alloy heart melt «ONLY» technology development. // Proc. of 8-th world titanium conference: «Titanium'95,» held in Birmingham (UK), Cambridge: University press.- 2 .-P. 1438-1453
- C.E. Shamblen, W.H. Buttrill, and G.B. Hunter Titanium Alloy Hearth Melt Technology Proc. of 7th world titanium conference Titanium92, Science and Technology held in San-Diego, (California, USA).
 Warrendale: TMS-AIME, 1992.- III. P. 2,451 -2,458
- 10. W. H. Buttrill, C. E. Shamblen and G. B. Hunter Hearth Melting of Titanium Alloys for Aircraft Engine Applications P. 209
- 11. Trefilov V.I., Minakov V.N., Polkin I.S., Kudryashov V.G. The effect of impurities of fracture in titanium alloys //Deutze Geselschaff fur Metallcunde E.V. Munchen. 1984.- V.3. P.1961.
- 12. Trefilov V.I., Minakov V.N., Turtsevich E.V., Grabin V.F., Kompan Ya.Yu. Electric slag welding of titanium alloys: engineering potential //Titanium metal science and technology (Proc. Of the 3rd Titanium int. Conference, held in Moskov 18-21 may 1976.) M. VILS. 1978. II P.339-343.
- 13. Trefilov V.I., Minakov V.N., Turtsevich E.V., Grabin V.F., at. all Cold brittleness temperature of titanium alloy VT22, welded by electric slag process// //Titanium metal science and technology (Proc. Of the 3rd Titanium int. Conference, held in Moskov 18-21 may 1976.) M. VILS. 1978. II P.?.
- 14. Minakov V.N., Turtsevich E.V. Perepelkin A.V., Kompan Ya.Yu. The influence of heat treatment on mechanical properties of high strength titanium alloys// In.: "Production and behaviour of materials in space", M., 1978. (in Russian)
- 15. Minakova A.V., Minakov V..N., Minakov N.V. Adeev V.M. The influence of the failure temperature of the MEM and IEBM titanium on the dimple size. // Met. Phys Adv. Tech., 1995. 17(11). P. 71-77
- 16. Tichonovskiy A.L., N.K.Lashuk, A.A. Tur, at all. Electron beam melting of titanium sponge using high voltage glow discharge guns //Problems of SEM, 1991,№1 (in Russian
- 17. B.I. Medovar Information on ICVM-II Problem of SEM, 1992, No. C. 105 (In Russian)
- 18. S.M.Sindler "Development of titanium industry"// Non-ferrous metals, 1997, 7, P.82-85 (In Russian)
- 19. V.ILakomski, O.S.Zabarilo, and oth., "Production of secondary titanium by plasma-arc melting. Problem of SEM.-1976, 4. P. 94-96 (In Russian).
- Minakov V.N. Minakov N.V., Podrezov U.N. Technical grade titanium hardening features under the uniaxial stress conditions // Met. Phys Adv. Tech., 1998. 20(9). P. 72