

# We are IntechOpen, the world's leading publisher of Open Access books Built by scientists, for scientists

6,900

Open access books available

186,000

International authors and editors

200M

Downloads

Our authors are among the

154

Countries delivered to

TOP 1%

most cited scientists

12.2%

Contributors from top 500 universities



WEB OF SCIENCE™

Selection of our books indexed in the Book Citation Index  
in Web of Science™ Core Collection (BKCI)

Interested in publishing with us?  
Contact [book.department@intechopen.com](mailto:book.department@intechopen.com)

Numbers displayed above are based on latest data collected.  
For more information visit [www.intechopen.com](http://www.intechopen.com)



## Genesis of Gas Containing Defects in Cast Titanium Parts

Vladimir Vykhodets<sup>1</sup>, Tatiana Kurennykh<sup>2</sup> and Nataliya Tarenkova<sup>2</sup>

<sup>1</sup>*Institute of Metal Physics, Ural Division, Russian Academy of Sciences,*

<sup>2</sup>*VSMPO-AVISMA Corporation,  
Russia*

### 1. Introduction

At present, gas-containing inclusions of metallurgical origin like Ti-O, Ti-N, and Ti-O-N are one of the main problems of titanium production. These defects negatively influence the mechanical properties of titanium alloys, as the mechanical properties (hardness and plasticity) of gas-containing inclusions differ appreciably from the corresponding characteristics of alloy matrices. Therefore, cracks nucleate in defect-matrix contact zone upon loading, which results in catastrophic fracture of heavily loaded parts. For example, the tragic consequences of failures of aircraft engine parts owing to the presence of similar effects are well known. The technology of vacuum arc remelting (VAR) of titanium alloys is most vulnerable from the view point of the formation of gas-containing defects, and, at the same time, it dominates in the overall production of titanium alloys. Efforts over many years have not resulted in the development of a technology for the defect-free production of titanium alloys by the VAR method and the titanium defect problem known also as “hard alpha” remains a troublesome aspect of the use of titanium alloys. Attempts to remove or modify the hard-alpha inclusions by diffusion homogenization of ingots had failed due to the long time required for the appropriate heat treatment at high temperature. Therefore, the genesis of defects under the conditions of industrial processes, their identification in each specific case, classification, and the elaboration of measures for reducing the formation of defects are topical questions.

A generally accepted concept has been developed (Bellot & Mitchel, 1994), according to which the sources of defects are particles of titanium and its alloys with a high concentration of oxygen and nitrogen atoms (the predominant element is nitrogen) and liquidus temperatures exceeding the smelting process temperature. Such particles can be formed at different stages of the preparation of batch materials for smelting. It may be supposed that all the components of a batch, such as titanium sponge, titanium return production wastes, and titanium containing alloy additions, are potential sources of defects. Smelting products with rather high concentrations of oxygen and nitrogen atoms may also be sources of defects. In melting oxygen and nitrogen atoms pass into a melt due to diffusion, the melting temperature of particles enriched in light elements decreases, the inclusion size decreases too, and conditions for the their dissolution are created. This scenario takes place for most particles enriched in oxygen and nitrogen atoms. Nevertheless, some of them remain solid by the end of melting

although titanium sponge (plus titanium return production wastes, and alloy additions) are melted twice for many applications and even three times for critical aeroengine parts.

Studies that have been conducted on the defects in titanium alloys may be conditionally classified into two groups: works with model defect sources and model smelting processes and works based on the analysis of the characteristics of defects revealed for industrial processes. At present, the model approach is predominant. However, the patterns established in model experiments may be not adequate for the processes that occur during smelting under industrial conditions, owing to the difference between the characteristics of real and model defect sources, industrial and experimental smelting modes, and also of the corresponding equipment.

In our opinion, the possibilities of the model approaches have been almost exhausted. This is shown by the fact that the model approaches has not led to substantial progress in the understanding of the genesis of defect formation under industrial conditions and the development of technologies for the defect-free production of titanium alloys. In this connection, we place our emphasis on gaining information on the processes of defect formation exclusively on the basis of the data characterizing industrial processes in the present work. In this case, it is impossible to study a statistically representative sample of defect sources, as, on average, a single defect is formed in several millions of particles of batch materials, so the study of a statistically significant ensemble of defects of industrial origin was used as the basis of this approach. This was achieved by the long term monitoring of a process in which the VAR technology was used. We know only one work of a similar type in the literature. This is a report that was published by the Material Development Department of the TRW Company (Grala, 1968). Metallographic and micro X-ray spectral studies of seventeen metallurgical defects in a Ti-6Al-4V alloy were performed in this work. According to the results of this work, the formation of defects may be caused by an increased concentration of atoms of both light elements (oxygen and nitrogen) and aluminum.

## 2. Materials and methods

The detection of inclusions in metal and their localization in semi products (rods, billets, plates, and sheets) made from titanium alloys was conducted by the method of ultrasonic testing. After the device received an echo signal indicating the presence of an internal defect we marked and cut out a specimen containing the zone of the signal with increased amplitude, whereupon specimens were subjected to mechanical processing (layer by layer metal stripping) accompanied by metallographic studies. As a result, the zone of the maximum echo signal was brought into the visible plane of a metallographic polished specimen. These polished specimens were the objects of metallographic studies; measurements of the microhardness and the concentration of oxygen, nitrogen, and alloying metal atoms in the matrix and the material of defects were also made.

The measurements of the microhardness in the zone of defects were performed with the use of a device with a Vickers diamond tip. The load of the indenter was 200 g. The concentrations of the atoms of alloying metals (mainly, aluminum and vanadium) in revealed defects were determined by X-ray spectral analysis using the energy dispersion method on an EDAX scanning electron microscope. The statistical error in the measurement of the aluminum concentration was 10% of the measured value for aluminum

concentrations of less than 5.0% (hereinafter, all concentrations are given in weight percents). For higher aluminum concentrations, the error was 0.5% and the error in the measurement of the vanadium concentration was approximately 0.5%.

The concentrations of oxygen and nitrogen atoms in specimens were determined by nuclear microanalysis technique (NRA) on a 2 MV Van de Graaf accelerator with the use of the  $^{16}\text{O}(d,p_1)^{17}\text{O}$  and  $^{14}\text{N}(d,\alpha_1)^{12}\text{C}$  reactions at an incident beam particle energy of 0.9 MeV. The method in its traditional variant (Vykhodets et al., 1987), in which the diameter of the incident beam of the accelerator was 1.0 or 2.0 mm, was used for the measurement of the average concentrations of light element atoms in a matrix of the titanium alloys. Defects usually had smaller sizes. In this connection, we developed a variant of the NRA method with enhanced locality (Vykhodets et al., 2006) to study them. Using this method, we measured the average concentrations of oxygen and nitrogen in defects 0.1–2.0 mm in size. In this case, each specimen that was measured was equipped with an individual collimator. Its dimension was chosen with consideration for the size of an analyzed zone, i.e., a defect, in a certain specimen. A scheme of NRA experiment is shown in Fig. 1.

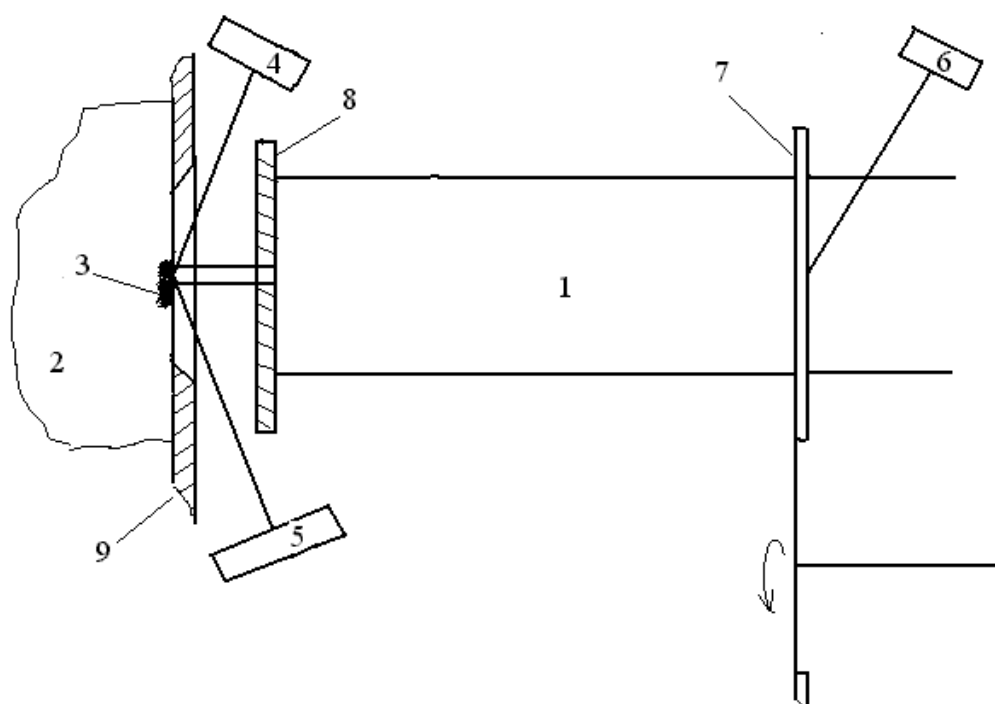


Fig. 1. Scheme of NRA experiment: 1 – incident beam; 2 – specimen; 3 – defect to be investigated; 4,6 – silicon surface-barrier detectors of the system of monitoring incident beam; 5 – silicon surface-barrier detector, which register spectrum of the products of nuclear reactions for the studied specimen; 7 – propeller of the system of monitoring incident beam; 8 – individual collimator; 9 – plate with specimens.

Using the NRA method, we measured the concentrations without destruction of a specimen down to a depth of nearly 2.0  $\mu\text{m}$ . This allowed us to distinguish the effects of the volume and surface (uncontrollable) alloying of alloys with the atoms of light elements in the nondestructive profile analysis. These appeared, in particular, at the stage of the preparing

polished specimens and could propagate in a specimen down to a depth of nearly 0.5  $\mu\text{m}$ . The effects of the surface alloying of specimens with atoms of oxygen and nitrogen are excluded from consideration in all the results below. In this work, the sensitivity in the measurements of the concentrations of oxygen and nitrogen was approximately 0.01%, the statistical error in the measurements of concentrations was at a level of several percent of a measured value. The mounting of the individual collimators 8 and the specimens 2 with the defects onto the plate 9 was performed under an optical microscope before the plate was placed into the vacuum chamber of the accelerator unit. As a result, the precision with which the beam hit the defect was 0.01 mm. The typical spectrum of nuclear reactions is shown in Fig. 2.

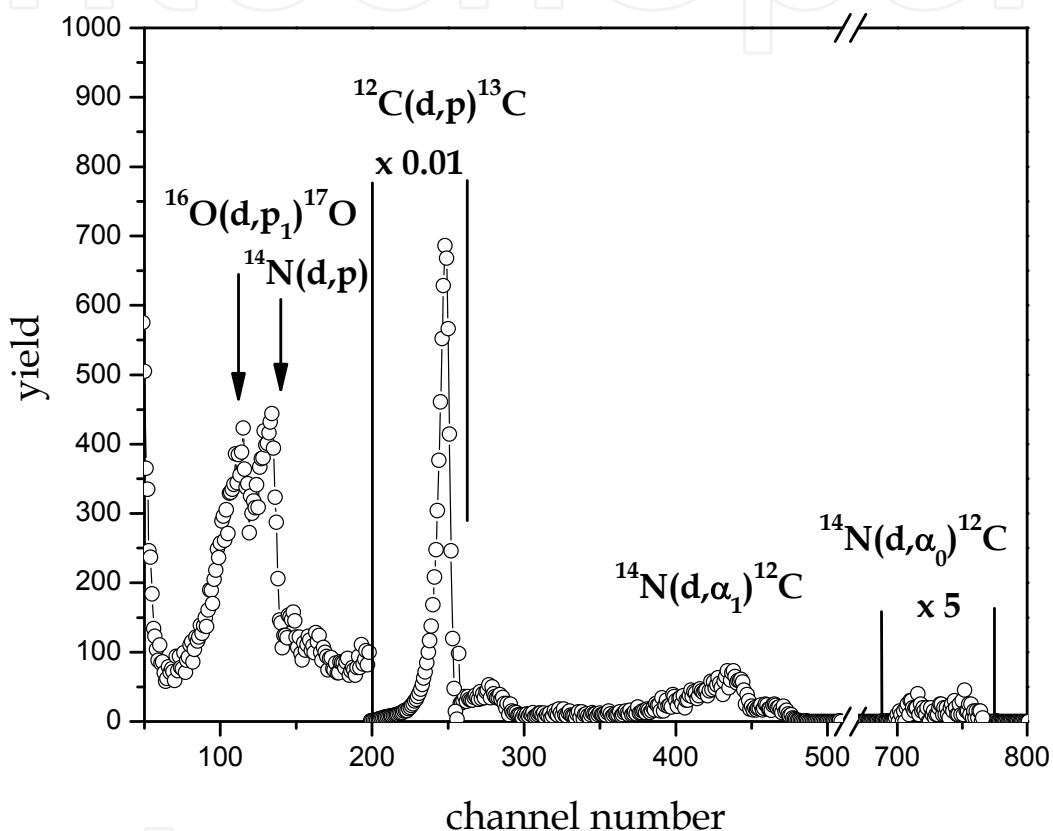


Fig. 2. The typical spectrum of nuclear reactions.

In total, more than 100 metallurgical defects revealed in the Ti-6Al-4V alloy were studied. This grade dominates in the overall production of titanium alloys. Defects revealed in other grades of titanium alloys were also investigated. In all the cases, the alloys were obtained by the VAR. Inclusions varied in their shapes and sizes and, to a first approximation, it was possible to distinguish spherical and extended defects. The causes of such variations have not been established as yet and this question is not discussed in this work. The minimum size of the studied effect was predominantly within the interval from 0.1 to 2.0 mm. The length of extended defects could amount to several centimeters. The full range of studies was conducted not only for defects, but also for the matrix surrounding a revealed defect. These investigations were performed for verification, and their results did not reveal any particular features. The microstructure, microhardness, and the concentrations of gaseous impurities and metal atoms near defects were identical to those for the zones remote from

defects. Some of the results represented in this work were previously published in (Tarenkova et al., 2006; Vykhodets et al., 2007; Vykhodets et al., 2011), but the corresponding data were obtained on an appreciably smaller sample of defects.

The above mentioned characteristics of defects (concentrations of gaseous impurities and aluminum, microhardness etc.) are traditional in the description of defects in titanium alloys. In this work, we included also the data on defect coordinates in ingots into the defect database. They are also important for the understanding of the genesis of gas-containing defects since in the industrial VAR technology the rate of dissolution of defect sources in different ingot parts may differ essentially. This is connected with different temperature of the liquid pool in different parts of the ingot and with the use of magnetic stirring in the VAR technology. This operation results in different liquid flow velocity around solid defect sources in the melt. Earlier, in model experiments (Bellot et al., 1997; Reddy, 1990; Schwartz, 1993; Mitchell, 1984) it was found that the dissolution rate of defect sources strongly depends both on the temperature of the liquid pool and the degree of stirring. For example, the measured dissolution rate of nitrogen-containing inclusions for intense stirring was 10 times faster than those without stirring and the dissolution rate doubled for the temperature rise of about 100°C. In our study, the data on the distribution of defects in ingots were obtained by means of the following procedure. Under production conditions, for each metal volume we made a flow chart of technological operations. After detecting the defect in semiproduct or finished product, the flow chart allowed finding the position of the metal volume in the ingot from which the semiproduct or finished product was produced. Further calculations allowed determining two coordinates of a defect in the ingot: distances from a defect to the ingot base and the ingot axis. Accuracy of coordinate determination in the ingots was about several millimeters.

### 3. Experimental results and discussion

#### 3.1 Oxygen and nitrogen concentration in defects

Histograms of the distributions of defects over the concentrations of nitrogen and oxygen are shown in Figs. 3 and 4. Almost all the revealed defects contained excess concentrations of oxygen and nitrogen in comparison with the volume of titanium alloys; the excess ranged from several times to several tens of times larger. In almost of cases, the concentration of oxygen was slightly higher than that of nitrogen. At the registered concentration level of gaseous impurities, the alloying of inclusions by nitrogen is the predominant factor in the formation of defects. This is associated with the fact that the alloying of titanium by nitrogen leads to an appreciably higher increase in the liquidus temperature of a material in comparison with the alloying of titanium by oxygen. This result, which indicates the predominant role of nitrogen in the formation of gas-containing defects, agrees with the data from study (Wood, 1969). The data obtained on the concentration of gaseous impurities in defects turned out to be insufficient for the verification of the concept, according to which all sources of defects did not dissolve in the liquid pool because of a high concentration of nitrogen and oxygen in them. There are two main reasons for this. Firstly, the range of possible values of smelting temperatures for an industrial process is not known with certainty. Secondly, the concentration of gaseous impurities could not be measured strictly at the end of smelting since during ingot cooling or other high-temperature treatment processes the concentration of light element atoms decreased due to their diffusion from a



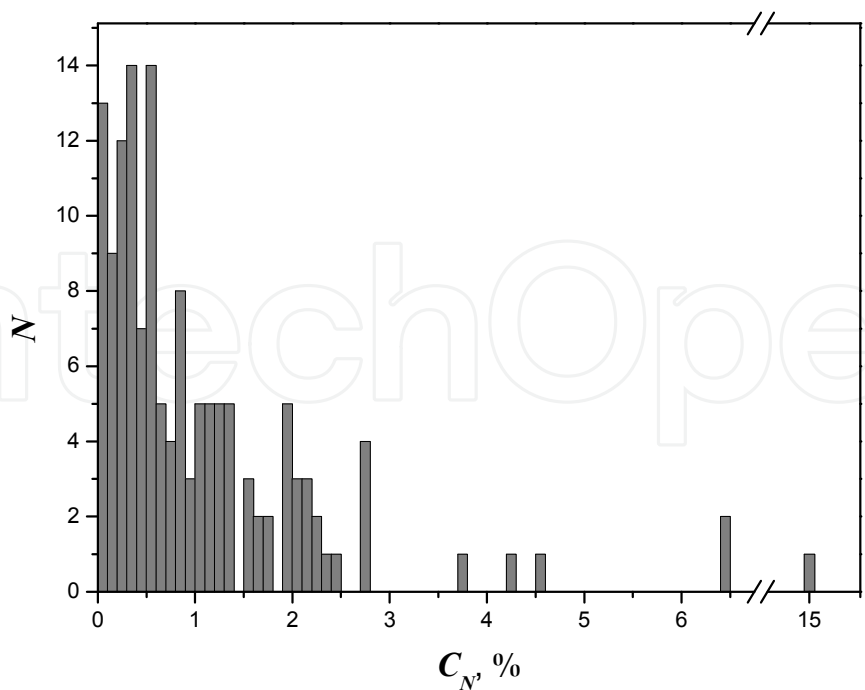


Fig. 3. Distribution of defects over the concentration of nitrogen in them:  $N$  is the number of defects;  $C_N$  is the average concentration of nitrogen in a defect.

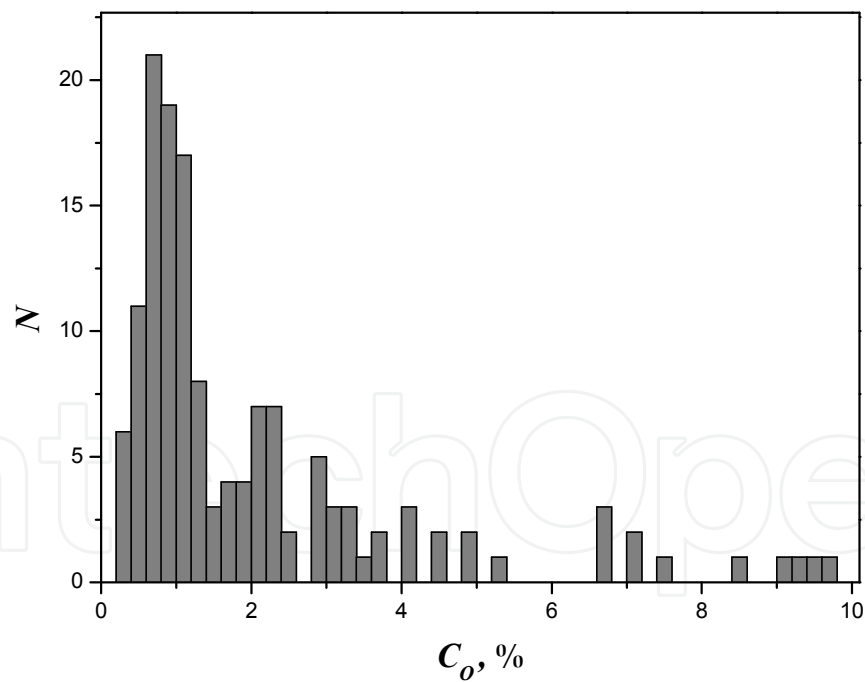


Fig. 4. Distribution of defects over the concentration of oxygen in them:  $N$  is the number of defects;  $C_O$  is the average concentration of oxygen in a defect.

defect to a solid titanium alloy. Fig. 5 presents a calculated histogram of defect distribution on the liquidus temperature of the defect material. The liquidus temperatures  $T_L$  were found using the data of binary diagrams of states for the systems Ti-O and Ti-N (Bellot & mitchell, (1994); Fromm & Gebhardt, 1976). Here, an additive approximation was used, namely, it

was assumed that in Ti-N and Ti-O-N systems nitrogen atoms raise the liquidus temperature of the defect material by the same value if nitrogen concentrations are equal in these systems. The same additive approximation was used for Ti-O and Ti-O-N systems. The effect of vanadium and aluminum on the liquidus temperature of the defect material was not considered since it is much weaker than that of nitrogen and oxygen.

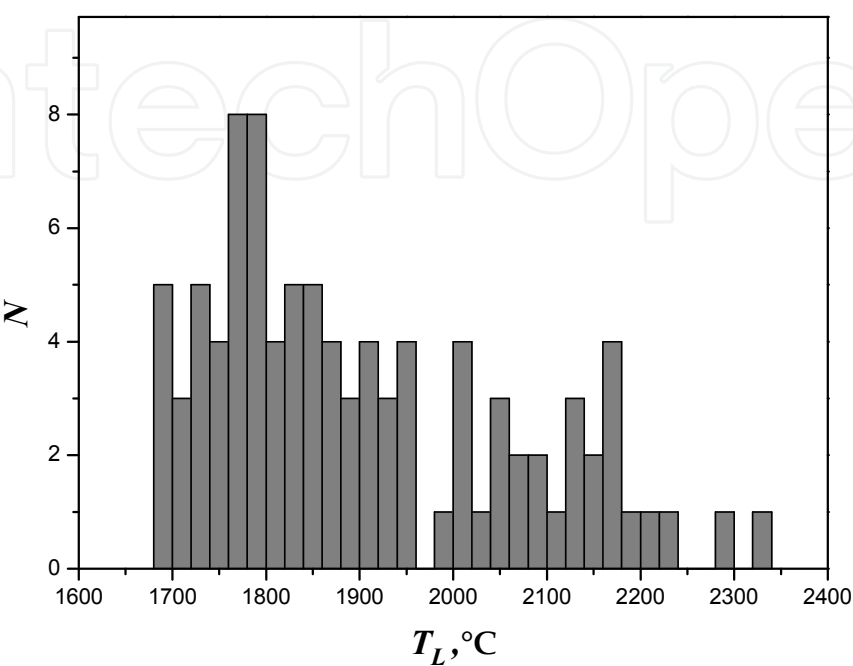


Fig. 5. Distribution of defects over the liquidus temperature of the defect material:  $N$  is the number of defects;  $T_L$  is the calculated liquidus temperature of the defect material.

The temperature of smelting is usually assumed to be close to 1900°C. In study (Bellot et al., 1997), a lower value (1780°C) is reported. Anyway, it can be stated that defects were revealed, the material of which has the liquidus temperature both higher and lower than that of the smelting process. This conclusion can hardly change the rough approximations used for the calculation of the liquidus temperature of the defect material. The existence of defects with liquidus temperatures below the temperature of smelting in principle can be due to diffusion processes occurring in the system upon completion of smelting.

On the whole, in most cases the concentration of nitrogen in defects was higher than in the alloy matrix. For such defects, the data on nitrogen and oxygen concentrations are not in conflict with the existing viewpoint that the sources of defects are titanium particles and its alloys with high concentrations of oxygen and nitrogen. At the same time, prolonged monitoring of the industrial technology revealed about ten defects, in which the concentration of nitrogen did not exceed that in the alloy matrix. Naturally, such defects cannot be considered as gas-containing defects; they turned out to be abnormal also in some other respects. We shall dwell on this question in greater detail in section 3.3.

3.2 Microhardness

Under the conditions of industrial production, the microhardness is traditionally measured in each case that a defect is revealed. These data are usually used as an



indicator of the presence of light element atoms in defects. In this study, we did not observe any univocal correlation between the microhardness and the concentration of gaseous impurities in defects. This can be seen from Fig. 6, which illustrates the dependence of the microhardness on the total concentration of oxygen and nitrogen in defects. For definiteness, in Fig. 6, we give only the minimum values of the microhardness in the body of a defect because of a slight scatter observed in the measurements of the microhardness.

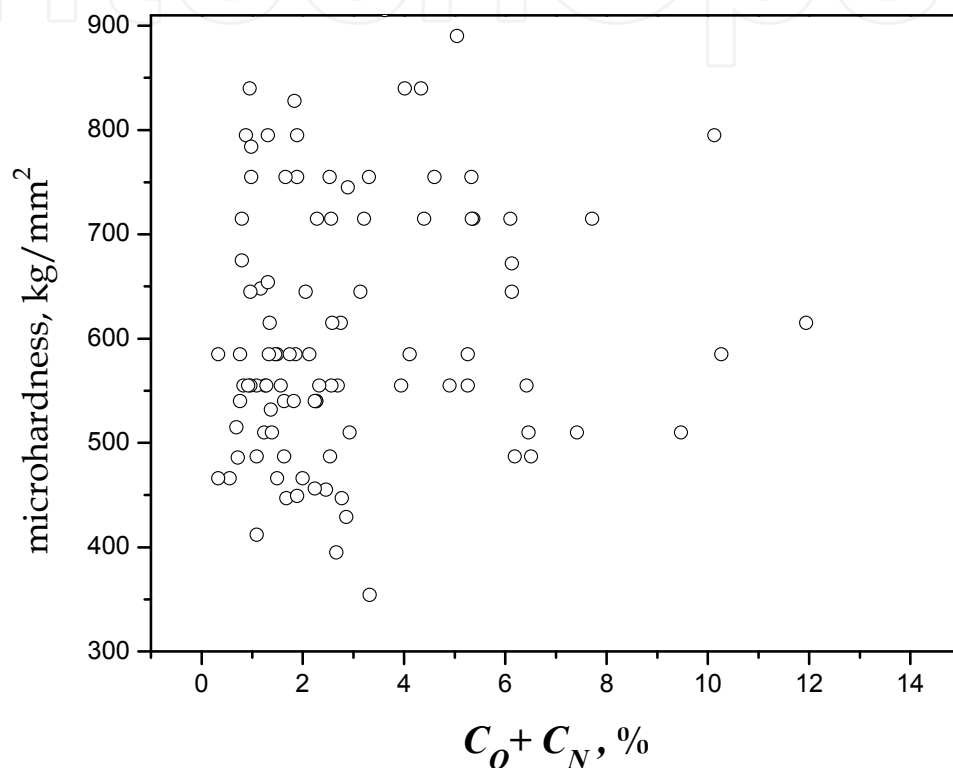


Fig. 6. Microhardness of defects versus total concentration of nitrogen and oxygen ( $C_O + C_N$ ) in them.

We also detected several defects with a very high microhardness from  $\sim 900$  to  $2600$  kg/mm<sup>2</sup>, the corresponding data are not shown in Fig. 6. These defects proved to be abnormal in several other respects. As in the case of the results on the concentration of nitrogen in defects, this circumstance will be used in this work for the classification of revealed defects. The result shown in Fig. 6 is not trivial. A linear or close to linear dependence of the microhardness on the concentration of gaseous impurities is usually observed (David et al., 1979). From Fig. 6, it follows that in the case of industrial defects, the value of microhardness cannot serve as a criterion of the presence of gaseous impurities in them. On the whole, the only pattern that can be noted with respect to the microhardness is that its value in defects is usually higher than that in the matrix (nearly  $350$  kg/mm<sup>2</sup>).

3.3 Aluminum concentration at the centre of defects

As different batch materials before smelting and smelted alloys contain various quantities of alloying elements, the patterns of the concentrations of alloying element atoms in defects may be used for the identification of defect sources. A similar idea was suggested in study (Bewlay & Gigliotti, 1997); this approach is widely used in industry. Let us estimate the prospects of this approach for the Ti-6Al-4V alloy. This alloy mainly contains atoms of aluminum (from 5.50 to 6.75%) and vanadium (from 3.50 to 4.50%).

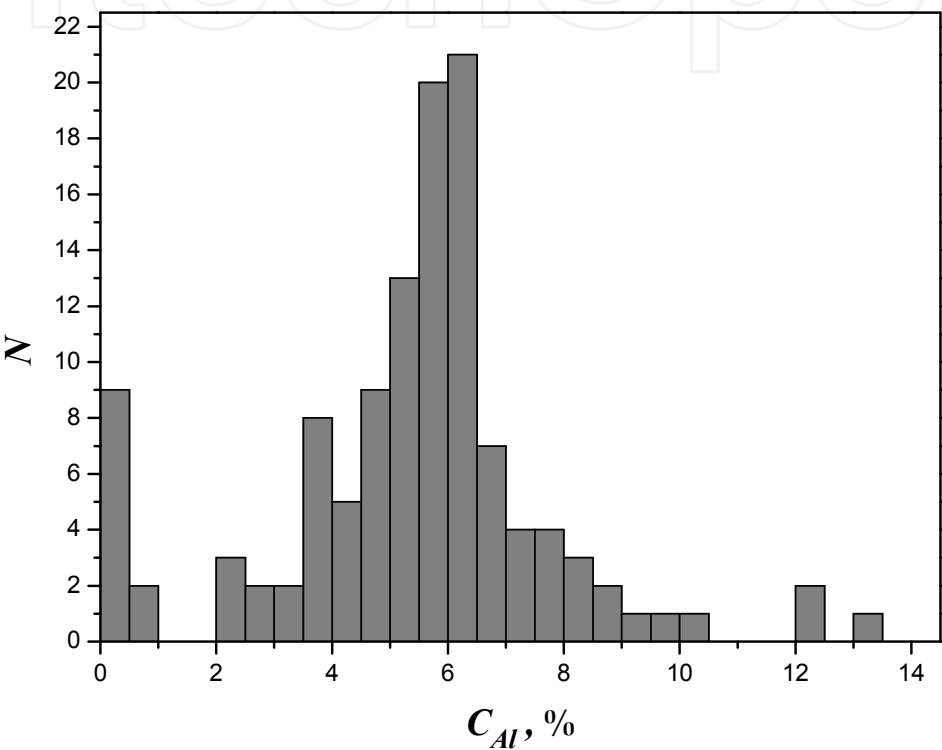


Fig. 7. Distribution of defects revealed in the Ti-6Al-4V alloy over the concentration of aluminum:  $N$  is the number of defects;  $C_{Al}$  is the aluminum concentration at the center of a defect.

Aluminum and vanadium are also present in the same quantities in titanium production wastes. Titanium sponge does not contain appreciable amounts of alloying elements; only atoms of aluminum (20.0%), oxygen, and titanium are present in the oxygen containing addition alloy before smelting. A much more difficult situation with the concentration of aluminum and vanadium takes place in the smelting products, whose particles can pass into a melt in the process of smelting. According to the data of several reports of the VSMPO-AVISMA Corporation, the concentration of vanadium and aluminum in smelting products obtained in the smelting of the Ti-6Al-4V alloy may be 2.0–4.2 and 4.7–20.0%, respectively. On the whole, it is possible to talk of higher aluminum concentrations in smelting products in comparison with those in a melted alloy.

The histogram of the distribution of defects over the aluminum concentration at their centers is shown in Fig. 7. The presence of a pronounced peak in the distribution may be

associated with the fact that all the probable defect sources fall within the corresponding concentration interval. It can be seen that the genesis of only 26 defects can reliably be established on the basis of the measurement results on aluminum concentration. These were defects with an aluminum concentration of less than 4.0%; their origination from titanium sponge does not raise any peculiar doubts. The remaining revealed defects cannot be identified with the use of the data shown in Fig. 7. The vanadium concentration at the center of defects was also measured. As shown by analysis, the data of vanadium do not clarify the situation at all, so they are not given here. Hence, we can state that the approach that is used in practice for the identification of defect sources and based on the measurement of the concentration of aluminum and vanadium in defects has no any substantial grounds. It is not inconceivable that progress in this matter may be made with the use of additional criteria. First of all, we may use the analysis of the dependence of the aluminum concentration at the center of a revealed defect on its linear size.

To analyze the diffusion processes that occur during the smelting of titanium alloys in the subsystem of aluminum atoms we shall use the solution of the second Fick equation for the diffusion from a finite sized body with conjunctive boundaries in the form (Fromm & Gebhardt, 1976)

$$\frac{C - C^0}{C^S - C^0} = A(Dt/l^2), \quad (1)$$

where  $C$  is the concentration of an impurity (aluminum in our case) at the center of an inclusion at the end of smelting;  $C^0$  is the initial concentration of an impurity in an inclusion;  $C^S$  is the concentration of an impurity at the interface between an inclusion and melt;  $A$  is the function of the parameter  $Dt/l^2$ ;  $D$ ,  $t$  and  $l$  are the diffusion coefficient of an impurity, the annealing time and the linear size of a specimen, respectively.

The precise dependence  $A(Dt/l^2)$  for a cylindrical specimen (defect in our case) is shown in Fig. 8 (curve). This dependence also has a similar shape for other regular bodies. Certainly, in the case of defect sources that appear in the smelting of titanium alloys, it is necessary to take into account the fact that their sizes are reduced in the process of smelting. Because of the absence of a precise solution of the second Fick equation in the case of variable dimensions of an inclusion, we shall obtain an approximate solution of the problem for the dependence of the aluminum concentration at the center of a defect, whose dimensions are varied in the process of smelting. The calculated dependences  $A(l)$  obtained at several certain values of the parameter  $Dt$  are illustrated in Fig. 8 (straight lines). It can be seen that the dependences  $A(l)$  are close to linear ones within the interval of the most appreciable changes, i.e.,

$$A(l) = A(0) - \eta l. \quad (2)$$

The points in one of the straight lines indicate the precise values of the function  $A(l)$ . It can be seen that the deviation of the points from a linear dependence is slight. In this case, expression (1) may be applied also for the variable size of a defect with the substitution of  $A$  for its average value obtained for the entire interval of changes during smelting.

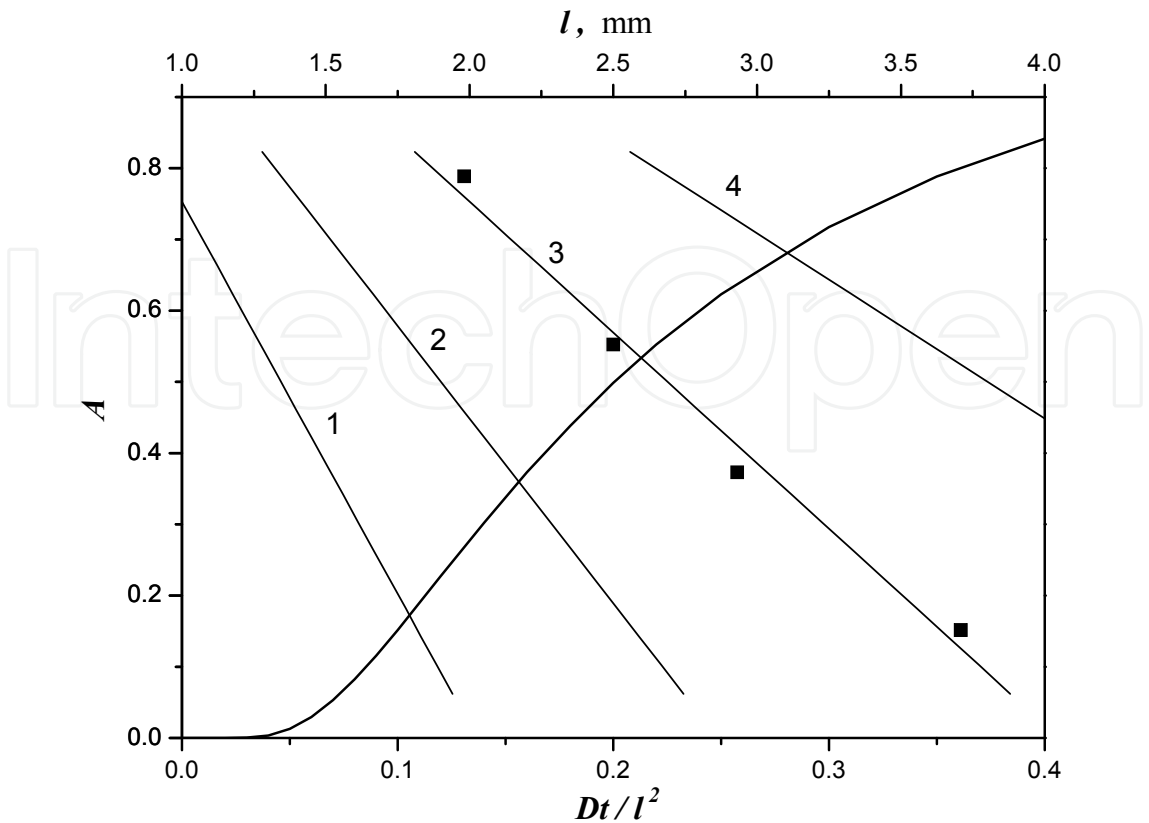


Fig. 8. Calculated dependences  $A(Dt/l^2)$  and  $A(l)$  for cylindrical specimens. The dependences  $A(l)$  were calculated at  $Dt$  of  $3.44 \cdot 10^{-5}$ ,  $6.88 \cdot 10^{-5}$ ,  $13.8 \cdot 10^{-5}$ , and  $27.5 \cdot 10^{-5}$  mm<sup>2</sup> (respectively: 1, 2, 3, 4).

Below, we will use the following notation:  $l$  is the radius of a cylindrical defect source before smelting,  $d$  is the diameter of a revealed defect, and  $\Delta$  is the thickness of the defect source layer dissolved in the process of smelting. Taking into consideration that

$$l = \frac{d}{2} + \Delta, \tag{3}$$

we obtain

$$C_{Al} = C^0 - (C^0 - C^S) \left[ A(0) - \frac{1}{2} \eta \Delta \right] + \frac{1}{2} (C^0 - C^S) \eta d \tag{4}$$

Hence, according to expression (4), the dependence of the aluminum concentration  $C_{Al}$  at the center of a revealed defect on its linear size  $d$  must be linear. It is self evident that this issue concerns the situation where the concentrations  $C^0$  and  $C^S$  are equal for all defect sources. Otherwise, a band of the values of the concentration  $C_{Al}$  must be observed in experiments. Equation (4) may be used for calculating the thickness  $\Delta$  of the defect source layer dissolved in the process of smelting. No fitted parameters are required to perform the corresponding calculations. As shown by the calculations, the value of  $A(0)$  does not depend on the parameter  $Dt$  and is determined only by the shape of a defect source. For example,  $A(0) \approx 1.49$  for a cylindrical specimen and  $A(0) \approx 1.31$  for a plate ( $l$  was accepted as the radius of a

cylinder or the half thickness of a plate). In this connection, the results of the calculations for the dissolved layer thickness  $\Delta$  depend on the shape of a defect. This creates certain problems, as defect sources can actually have any shape.

Note that the approach for obtaining the experimental data on the thickness  $\Delta$  of the defect source layer dissolved in the process of smelting and on the size  $l$  of a defect source before smelting for the industrial technology was first found in this work. Information of this kind is important for the development of methods that reveal the probable defect sources in batch materials. Moreover, the dissolution rate  $V$  of defect sources in the process of industrial smelting can be estimated in a linear approximation with the use of the value  $\Delta$  as

$$V = \frac{\Delta}{t}. \quad (5)$$

Previously, the defect dissolution rate, as mentioned in section 2, was determined only in model experiment (Bewlay & Gigliotti, 1997; Bellot et al., 1997; Reddy, 1990; Schwartz, 1993; Mitchel, 1984), and its strong dependence on the temperature of the liquid pool and the liquid flow velocity around solid particle was established.

Since reliable identification of the sources of 26 defects proved to be feasible, the additional identification method based on the analysis of the linear dependence of the aluminum concentration  $C_{Al}$  at the center of a defect on its size  $d$  (expression (4)) might be approved on the same defects. Actually, linear dependences (4) are expected to be fulfilled only qualitatively. This is connected with the following circumstances. To obtain such dependences, it was necessary to clarify the notion “linear size of a defect,” which is especially topical for extended defects. In specimens with complex shapes, the rate of diffusion processes is generally determined by the minimum linear size of a specimen, so it is chosen as the parameter  $d$ . Besides, the specificity of experiments performed in this work consisted in the fact that the concentrations of light elements and aluminum could not be measured at the end of smelting. During the cooling of ingots and other high temperature treatment processes, the atoms of impurities diffuse from defects into the solid titanium alloy and on the contrary, thus leading to a decrease or increase in the concentration of impurities in defects. In section 3.1 it was pointed out that the concentrations of oxygen and nitrogen in the revealed defects were apparently much smaller than those at the end of smelting. However, for aluminum this effect is expected to be appreciably weaker or negligible since the diffusion coefficients of aluminum in solid titanium are almost five orders of magnitude smaller than those of oxygen and nitrogen (Le Claire & Neumann, 1990; Le Claire, 1990). Besides, there are other reasons for the scatter of experimental data on  $C_{Al}(d)$ . In particular, parameters  $A(0)$ ,  $C^s$ , and  $C^0$  in expression (4) will vary within certain intervals for different defects and different smelting processes. For example, as already noted above, the concentration of aluminum in the melt varied in different smelting processes from 5.50 to 6.75%.

The experimental data on  $C_{Al}(d)$  for these 26 defects are illustrated in Fig. 9. It can be seen that there is no pattern in the corresponding data. The situation changes radically if defects with ordinary (below 800 kg/mm<sup>2</sup>) and high (above 900 kg/mm<sup>2</sup>) microhardness values are considered separately. As seen from Fig. 9, a linear dependence  $C_{Al}(d)$ , which results from the diffusion model, is observed for each individual group of defects in this case.

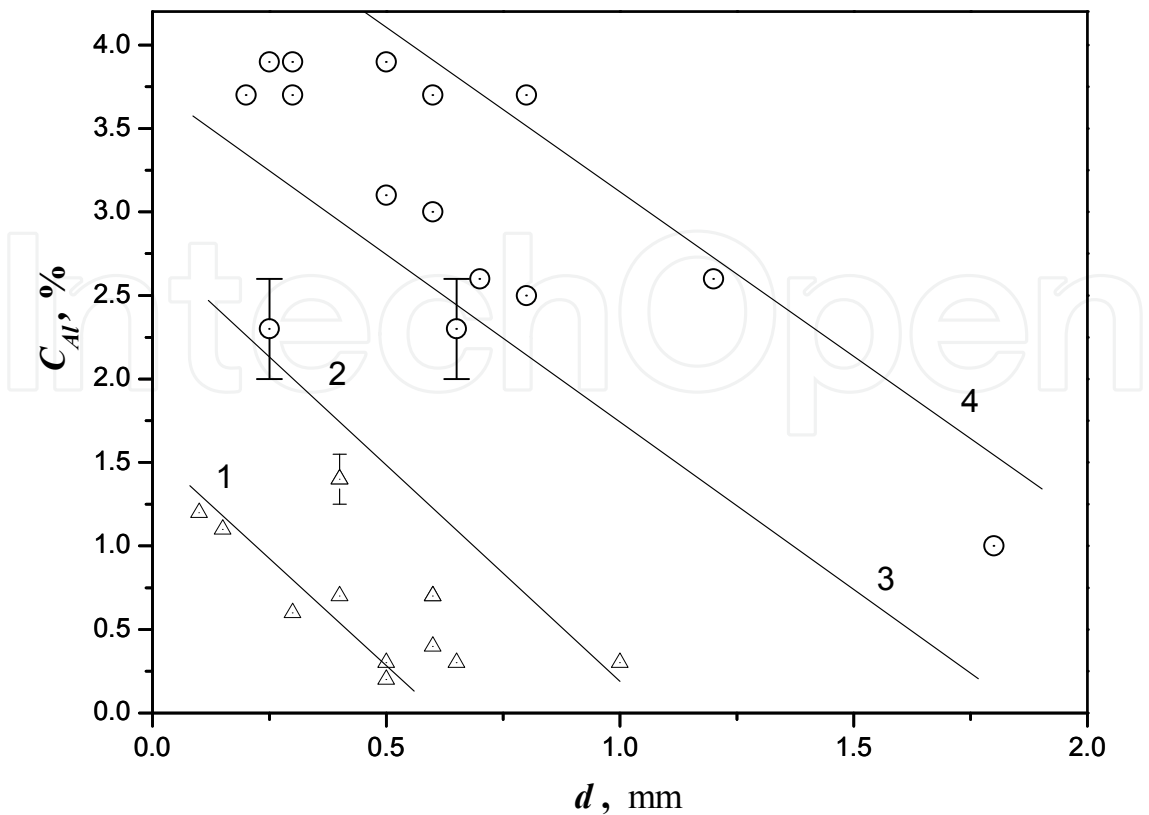


Fig. 9. The aluminum concentration  $C_{Al}$  at the centre of defects that were revealed in the Ti-6Al-4V alloy and originated from titanium sponge as a function of the minimum linear size of the defect: triangles are data for defects with microhardness above 900 kg/mm<sup>2</sup>; circles are data for defects with microhardness below 800 kg/mm<sup>2</sup>.

The concentration intervals for defects with ordinary and high microhardness are denoted in Fig. 9 as 1-2 and 3-4, respectively. It may be supposed that these two groups of defect sources differ from each other in the diffusion coefficients  $D$  of aluminum atoms in them (these coefficients are lower for defects with high microhardness). In turn, the difference between the values of  $D$  may be caused by different phase states of inclusions with high and ordinary microhardness values.

The results displayed in Fig. 9 are of interest mainly for two reasons. Firstly, they showed that the value of microhardness should be considered in the classification of defect sources. Secondly, the accuracy with which the theoretical linear dependence  $C_{Al}(d)$  was fulfilled proved to be high. This occurred in spite of a large number of factors determining the scatter of  $C_{Al}(d)$  data. The width of the concentration intervals denoted in Fig. 9 as 1-2 and 3-4 was 1.25%. This value agrees in particular with the technical specifications for the concentration range of aluminum in the Ti-6Al-4V alloy (from 5.50 to 6.75%). Note however that a single point in Fig. 8 falls outside of the established dependence  $C_{Al}(d)$ . The microhardness of this defect was about 800 kg/mm<sup>2</sup>,  $C_{Al} = 2.3\%$ , and  $d = 0.25$  mm. This shows that small and rare deviations from the theoretical dependence  $C_{Al}(d)$  can nevertheless take place.

In Fig. 10, the data on  $C_{Al}(d)$  are shown for almost all the defects revealed in the Ti-6Al-4V alloy. Only a single defect is not represented in this figure. Its minimum size (3.0 mm)



appreciably exceeded the sizes of the other defects, so it was unreasonable to provide the data on this defect. In connection with this exclusion, it should be noted that this defect was not inconsistent with the common patterns, which will be considered below. From Fig. 10, it can be seen that almost all the defects fit into the determined intervals of aluminum concentrations. In Fig. 10, they are denoted as 1–2, 3–4, 5–6, and 7–8. The first two intervals have already been discussed above. Interval 7–8, as well as intervals 1–2 and 3–4, correspond to the slanting linear dependence  $C_{Al}(d)$ . It makes sense to relate this to the sources of defects, in which the concentration of aluminum before smelting was higher in comparison with that in a melt. They might be the particles of smelting products and addition alloy. Interval 5–6 is horizontal. According to the diffusion model, it corresponds to the sources of defects in which the concentration of aluminum before smelting was close to that in the liquid pool. These may be particles of smelting products and return production wastes. The width of the concentration intervals was equal for all the corridors (1–2, 3–4, 5–6, and 7–8).

Let us discuss the sense of these results in connection with the problem of the identification of defect sources. First, we consider the case of small defects. From Fig. 10, it can be seen that such defects can be equally related to the intervals denoted as 3–4, 5–6, and 7–8. This means that in the case of small defects, their identification with the use of the discussed scheme is impossible, i.e., these defects may be formed from the particles of titanium sponge, return production wastes, smelting products, and addition alloy.

Further, let us consider medium and large sized defects. Here, the situation looks to be much more unambiguous. First, it is possible to note that only the defect sources that are characterized by a nearly equal and high aluminum concentration before smelting fitted in the interval 7–8. Such a condition is satisfied by the particles of addition alloy with an aluminum concentration of 20.0%. Hence, we can state that the defects that originate from the addition alloy are presented within interval 7–8. The absence of defects that formed from smelting products within the interval 7–8 and their presence within interval 5–6 seems to be unnatural. This allows us to think that defects that formed from smelting products are not represented within the interval 5–6, i.e., in the case of large defects, the 5–6 interval is caused by the formation of defects from return production wastes.

Let us consider the defects that did not fit in any of the intervals distinguished in Fig. 10. Only four similar defects were found. One of them had microhardness higher than 900 kg/mm<sup>2</sup>. The abnormal behavior of such defects has already been mentioned. The other three defects, whose data are denoted by asterisks in Fig. 10, have nitrogen concentrations that are lower than that in the matrix. It was previously mentioned that such inclusions cannot be reliably classified as gas-containing defects. We suppose that these defects were formed from the particles of smelting products and that their residence in a liquid pool might be very short, for which reason they fall outside of the diffusion patterns. Another defect, which is within the interval 5–6 in Fig. 10, should be classified with inclusions of the same type. The results for this defect are denoted by an asterisk. Consequently, it can be seen that all the cases in which the data on defects lie outside the distinguished intervals 1–2, 3–4, 5–6, and 7–8, are accompanied by abnormal properties of the defects (very high microhardness or very low nitrogen concentration).

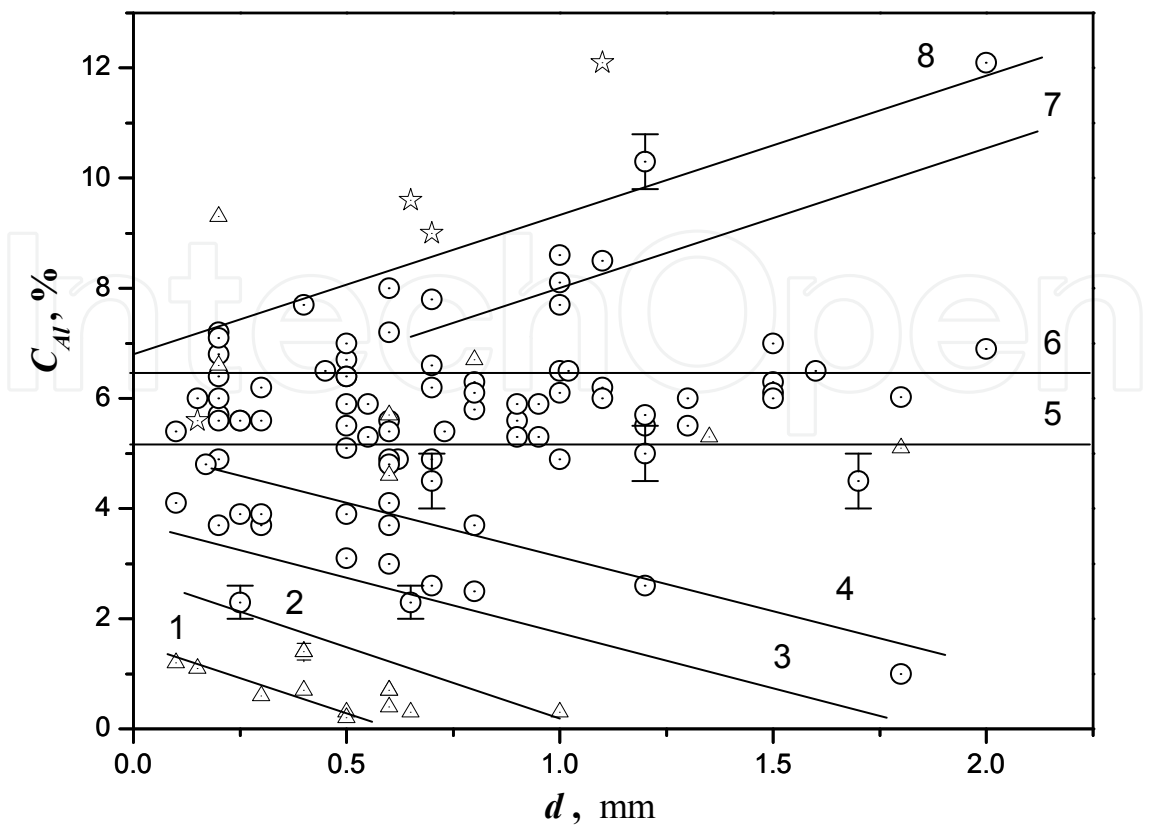


Fig. 10. The aluminum concentration  $C_{Al}$  at the centre of defects that were revealed in the Ti-6Al-4V alloy as a function of the minimum linear size of the defect: triangles are data for defects with microhardness above 900 kg/mm<sup>2</sup>; circles are data for defects with microhardness below 800 kg/mm<sup>2</sup>; asterisks are data for defects in which the nitrogen concentration does not exceed that in the matrix.

So, a method for the identification of the sources of defects in the industrial VAR technology was developed in this work. Diffusion laws form the basis of this method. This scheme has proven to be very simple for practical application. The identification of the source of a defect revealed in the smelting of the Ti-6Al-4V alloy requires a very limited data set containing the aluminum concentration at the center of the defect, its minimum size, the microhardness of the material of the defect, and the average nitrogen concentration in the body of the defect. Under industrial conditions, this information is usually available, so this method can be recommended for practical application. Note also that during the prolonged monitoring of an industrial process we did not observe defects whose data contradict this method.

Overall, the identification proved to be unfeasible for approximately 20% of the defects. According to the results of the identification, return production wastes were the source of approximately half of all the revealed defects, and nearly a quarter of them originated from titanium sponge. The remaining defects were formed from the particles of addition alloy and smelting products.

Further, using expressions (3)–(5) and the data shown in Fig. 10, the thickness of the defect source layer dissolved in the process of smelting and the dissolution rate  $V$  of defect sources were estimated. The average value of the thickness  $\Delta$  was about 2 – 3 mm. At the same time,

the data obtained do not exclude a broad interval of  $\Delta$  values during the industrial process: from 0 to 8.5 mm. With the corresponding estimates, the shape of the defects and the concentration of aluminum in the liquid pool varied from 5.50 to 6.75%. Besides, it was taken into consideration that the concentration of aluminum  $C^s$  at the boundary between the defect source and the melt can be smaller than the corresponding equilibrium concentration. With this approach, the interval of  $\Delta$  values was obviously overestimated. In reality, it will be smaller.

The average value of the dissolution rate  $V$  was approximately 1.0  $\mu\text{m/s}$ . The estimation was performed under the assumption that the residence time of any melt volume in the liquid phase was 2500 s. This value of the average dissolution rate is very low. Model experiments (Bellot et al., 1997) showed that the dissolution rate of the nitrogen-containing source of defects is 2.2, 4.0, and 6.7  $\mu\text{m/s}$  at the temperature of the liquid pool of 1700, 1800, and 1900°C, respectively, when the liquid flow velocity around solid particle in the liquid pool was 1 cm/s. Even when the liquid flow velocity around solid particle was 0.1 cm/s and the temperature was 1800°C, it was 2.0  $\mu\text{m/s}$ . One of the possible directions for defect formation reduction is enhancement of the dissolution rate of defect sources. The prospects of this trend will be discussed in section 3.4.

### 3.4 Location of defects in ingots

The experimental data on the location of defects in ingots were obtained for the ingots from 650 to 1000 mm in diameter and from 1200 to 2750 mm in height. Hereafter we use the following non-dimensional coordinates of defects:  $r/R$  ( $r$  is the distance from a defect to the ingot axis,  $R$  is the ingot radius) and  $h/H$  ( $h$  is the distance from a defect to the ingot base,  $H$  is the ingot height). The subjects of investigation were the distributions of defects over  $r/R$  and  $h/H$  coordinates, as well as the probability of formation of one, two, and more defects in an ingot. The experimental data on the location of defects in the ingots are presented in Figs. 11, 12 and 13. In Fig. 14, the number of defects revealed in the unit of the ingot volume is shown as a function of the non-dimensional radius  $r/R$ . The data for Fig. 14 were obtained by calculation from primarily data displayed in Fig. 13.

In a real process, the defect formation probability is  $p = N/N_h \ll 1$ , where  $N$  and  $N_h$  are the numbers of defects and heats, respectively. If the formation of a defect is an independent event the probability of formation of several defects in one ingot  $p(n)$  is

$$p(n) = p^n, \quad (6)$$

where  $n$  is the number of defects in the ingot. Since the formation of a defect is a very rare event ( $p \ll 1$ ), the formation of two or more defects in one ingot in this mechanism is almost impossible. However, experimental results indicated that one, two and even more defects can exist in ingots at the close probability.

Altogether we have discovered 205 defects in 86 ingots. 42 ingots contained one defect, 27 ingots contained two defects, 7 ingots had three defects, and in 10 ingots there were more than three defects. These results show that generally the formation of each defect cannot be considered as an independent event.

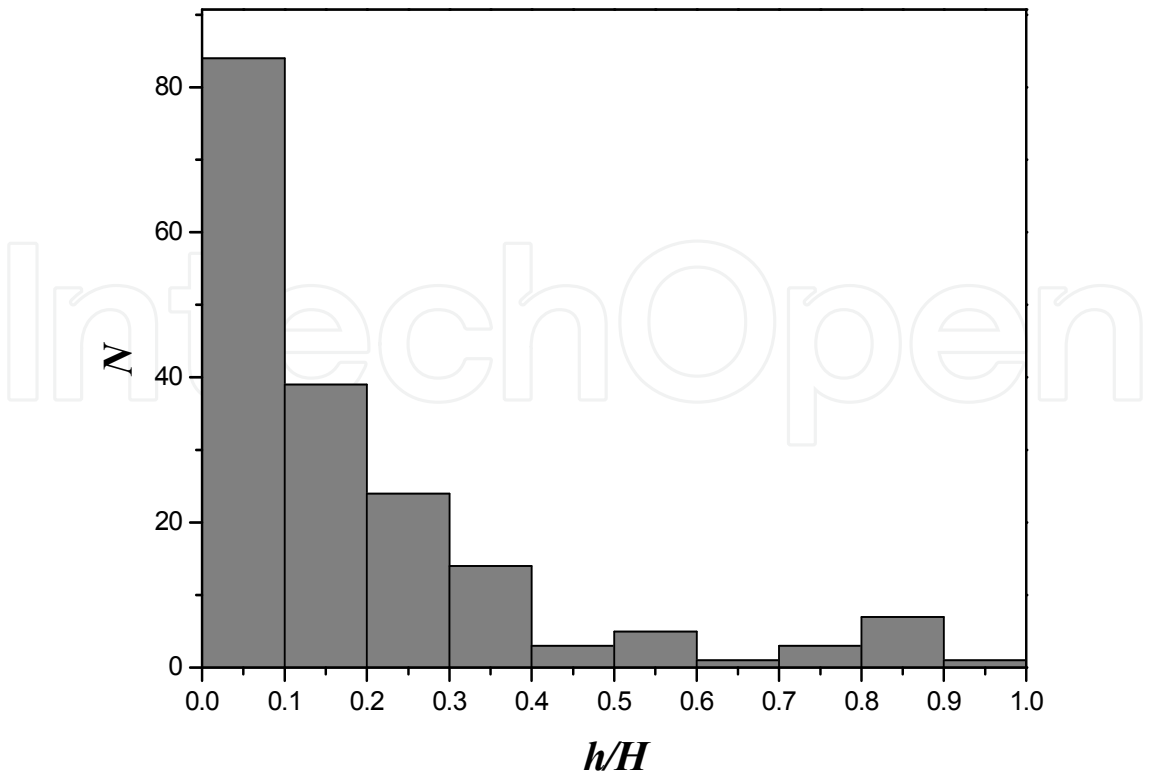


Fig. 11. Distribution of defects over their non-dimensional coordinate  $h/H$ :  $N$  is the number of defects;  $h$  is the distance from a defect to the ingot base;  $H$  is the ingot height.

For those cases when two defects were revealed in an ingot simultaneously, we analyzed the differences in  $h$  and  $r$  coordinates of these defects. In the majority of cases, such defects were very close to each other (the difference in  $h$  and  $r$  coordinates usually did not exceed several millimeters). Only in one case the difference in the  $h$  coordinates was very large (more than 600 mm). Based on this result we can assert that the sources of defects usually do not move over large distances during smelting.

From Figs. 11 and 14 it is seen that the distribution of defects over the ingot height and radius is strongly inhomogeneous. The defects are located chiefly near the ingot base and axis. Since the sources of defects do not move over large distances during smelting as mentioned above, it may be stated that the dissolution rate of defect sources near the ingot base and the axis is much smaller than the average dissolution rate in the ingot. This may be due to a decreased temperature of the liquid pool and (or) a decreased liquid flow velocity around solid particle in the melt. The ingot volumes with  $h/H$  values from 0 to 0.2 and  $r/R$  from 0 to 0.1 turned out to be most critical for defect formation. In this range of  $h/H$  values (20% of the metal volume) 68% of defects were revealed, while in the above range of  $r/R$  values (1% of the metal volume) there were revealed 17% of defects. These findings are indicative of strong inhomogeneity of defect distribution in the ingots. The number of defects in the unit volume of the metal for the volume bounded by  $h/H$  values from 0 to 0.2 and  $r/R$  values from 0 to 0.1 (0.2% of the ingot volume) turned out to be almost 60 times higher than the average value in the ingots.

Relying on these findings we can propose two mechanisms of defect formation. One of them suggests fluctuations in the characteristics of defect sources, i.e. the sources of defects

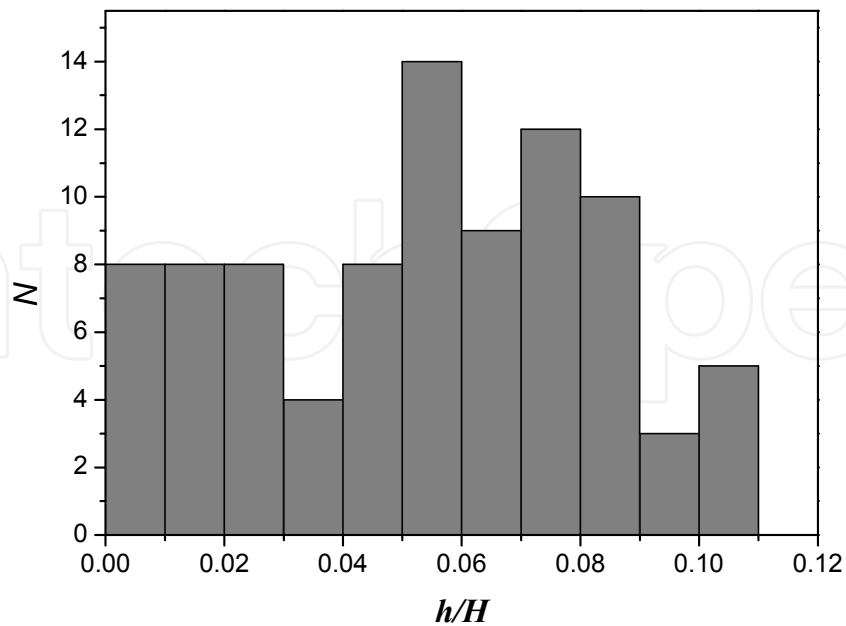


Fig. 12. Distribution of defects over their coordinate  $h/H$  near the ingot base:  $N$  is the number of defects;  $h$  is the distance from a defect to the ingot base;  $H$  is the ingot height.

contained in the charge materials have unfavorable characteristics for complete dissolution of defect sources during smelting. Such characteristics can include large sizes of defect sources and/or high concentrations of gaseous impurities in them. According to this mechanism two or more defects could form in one ingot and in one place of the ingot because some defect sources fell into two, three or more fragments during smelting. For this mechanism it is difficult to estimate the probabilities  $p(n)$  theoretically.

The second mechanism suggests fluctuations in the dissolution rate of defect sources during smelting. Such fluctuations can be associated with the existence of microvolumes with a decreased temperature of the liquid pool and/or microvolumes with a decreased liquid flow velocity around solid particles in the melt during smelting. According to this mechanism the formation of several defects in one ingot and in one place of the ingot is due to the value of fluctuation for the dissolution rate of defect sources. For this mechanism it is also difficult to determine theoretically the probabilities  $p(n)$ . Thus, there are no available data at present to give preference to one of the considered fluctuation mechanisms. At the same time, this question is a topical problem. Its solution determines the direction of the main efforts for reducing defect formation in the VAR technology. The first mechanism suggests scientific and technological measures for enhancing the quality of charge materials. With the second mechanism, efforts should be focused on the stabilization of the smelting regime. The results obtained on the distribution of defects over the ingot height (Fig. 11) agree with the reasoning (Bellot & Mitchel, 1994) that the temperature of the liquid pool is minimal near the ingot base. Probably, this cause for enhanced defect formation in the VAR technology cannot be eliminated. The results obtained on the distribution of defects over the ingot radius agree

with the model experiments (Bellot et al., 1997) on the examination of the dissolution rate of gas-containing inclusions as a function of liquid flow velocity around solid particles in the melt. In the existing technology, the rotation axis of the magnetic field coincides with the axis of the ingot during the whole period of smelting. This is likely to be one of the reasons why more defects are formed near the ingot axis than at the ingot periphery. Therefore, a possible measure for defect formation reduction is variation of the position of the magnetic field rotation axis during smelting. The estimates showed that with the use of this technique the defect formation can be reduced not more than in 2.5 times. In the estimation we used the data of Fig. 13 and Fig. 14. Besides, the following concepts were accepted.

First, different liquid flow velocities around solid particles in the liquid pool was considered to be the only reason for inhomogeneous distribution of defects over the ingot radius. Second, it was postulated that variation of the position of the magnetic field rotation axis can provide a high liquid flow velocity around solid particles in all parts of the ingot.

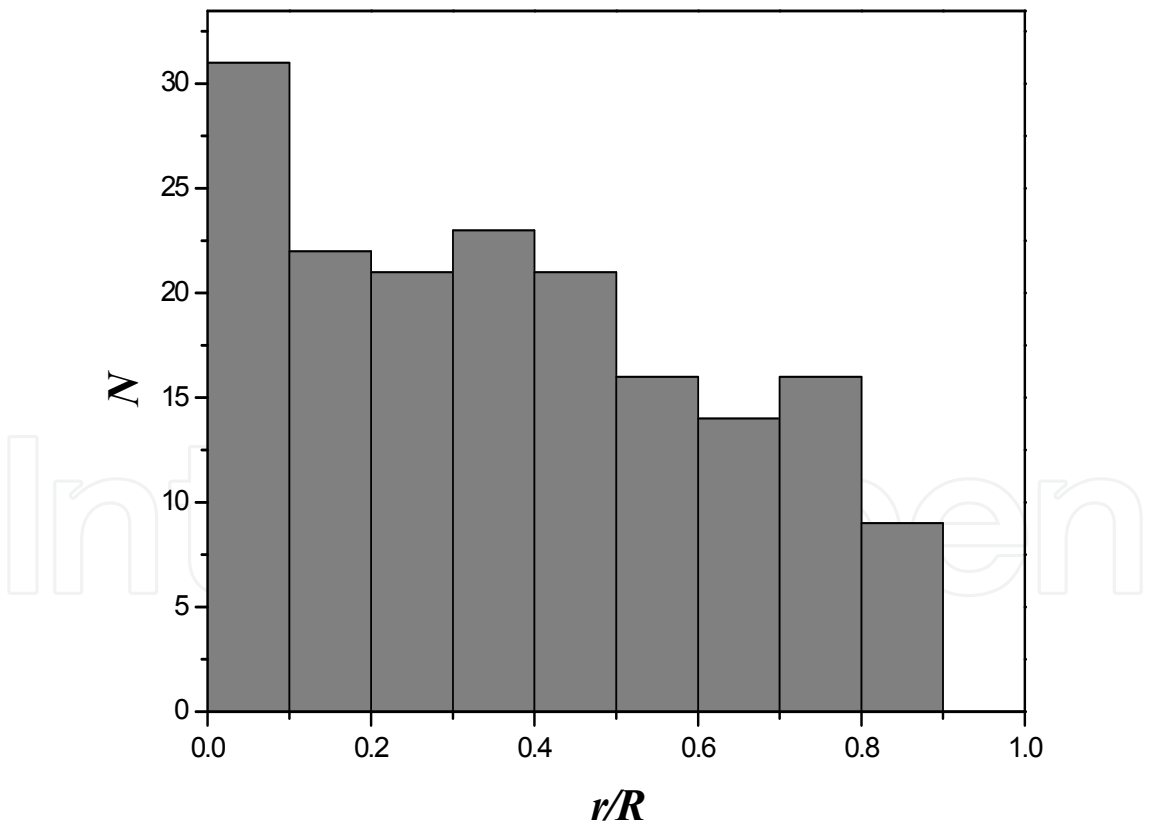


Fig. 13. Distribution of defects over their non-dimensional coordinate  $r/R$ :  $N$  is the number of defects;  $r$  is the distance from a defect to the ingot axis;  $R$  is the ingot radius.



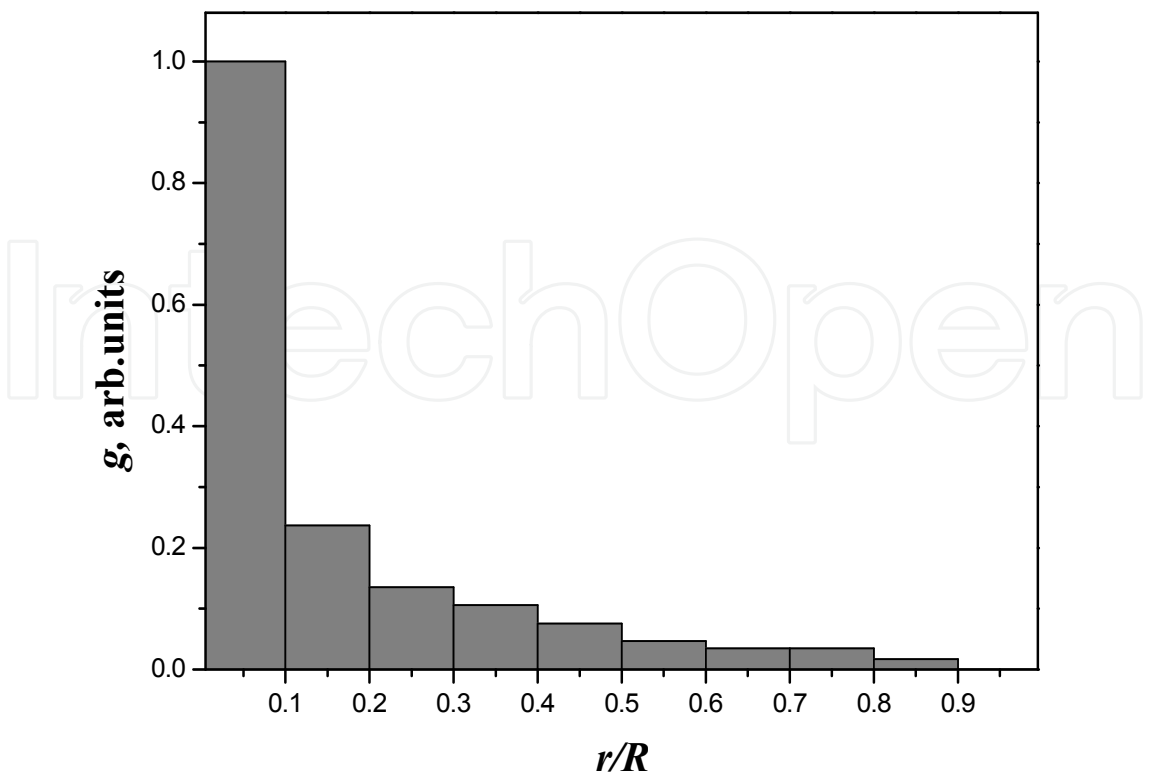


Fig. 14. Distribution of defects over their non-dimensional coordinate  $r/R$ :  $g$  is the number of defects in the unit of the ingot volume;  $N$  is the number of defects;  $r$  is the distance from a defect to the ingot axis;  $R$  is the ingot radius.

4. Conclusion

A database on the characteristics of metallurgical gas-containing defects in titanium alloys was obtained. This was achieved by the long term monitoring of a industrial process in which the vacuum arc remelting (VAR) technology was used. It is the most complete one that is available at present. It contains data on location of defects in ingots, the sizes of defects, their microhardness, and the concentrations of nitrogen, oxygen, and aluminum in them. Almost all the data are related to the Ti-6Al-4V alloy.

Almost all the revealed defects contained excess concentrations of oxygen and nitrogen in comparison with the matrix of titanium alloys; the excess ranged from several times to several tens of times larger. In almost of cases, the concentration of oxygen was slightly higher than that of nitrogen. At the registered concentration level of gaseous impurities, the alloying of inclusions by nitrogen is the predominant factor in the formation of defects. On the whole, the data on the concentrations of nitrogen and oxygen in the defects agree with a generally accepted concept, according to which the sources of defects are particles of titanium and its alloys with a high concentration of oxygen and nitrogen atoms.

It was shown that the approach that was used in practice for the identification of defect sources based on the measurement of the concentration of aluminum and vanadium in defects has no any substantial grounds.

The distribution of defects over the height and radius of ingots was studied; it turned out to be strongly inhomogeneous. The defects were located predominately near the ingot base and the ingot axis. The number of defects in the unit volume of the metal in the lower part and near the axis of the ingots was almost 60 times greater than the average value in the ingots. The inhomogeneous distribution of defects in the ingots is due to different dissolution rate of defect sources during smelting in different parts of the ingot. In turn, this results from a low temperature of the liquid pool in the lower part of the ingots and a low liquid flow velocity around solid particles near the ingot axis.

A large number of cases have been registered when not one but two, three or even more defects are formed simultaneously in the ingot. The probabilities of formation of one and several defects were comparable. Usually, several defects in the ingot were located very close to each other. These findings indicate that generally the formation of each defect cannot be considered as an independent event.

A method for identification of the sources of metallurgical defects in the smelting of titanium alloys has been developed. It is based on the diffusion mechanism for the modification of defect sources in smelting and the established dependences of the aluminum concentration at the center of a defect on its size. The identification scheme included data on microhardness and the average concentration of gaseous impurities in the zone of a defect. The efficiency of identification using this method was 80%.

## 5. Acknowledgment

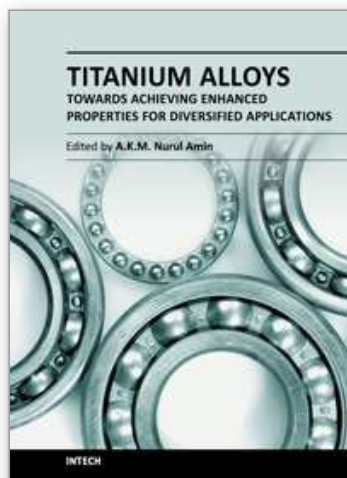
This work was supported by the project No 11-2-06-AVI of the Basic Research Program of the Ural Division of Russian Academy of Sciences "Identification of stages which are responsible for formation of gas-containing metallurgical defects for the industrial VAR technology of titanium alloys".

## 6. References

- Bellot, J.P. & Mitchell, A. (1994). Hard-alpha particle behaviour in a titanium alloy liquid pool. In: *Light Metals*, U. Mannweiler (Ed.), 1.187-1.193, The Minerals, Metals & Materials Society, ISBN 0-87339-264-7
- Bellot, J.P. et al. (1997). Dissolution of Hard-Alpha Inclusions in Liquid Titanium Alloys, *Metallurgical and Materials Transactions B*, Vol.28b, pp. 1001 -1010, ISSN 1073-5615 (print version)
- Bewlay, B.P. & Gigliotti, M.F.X. (1997). Dissolution rate measurements of TiN in Ti6242, *Acta Mater.*, Vol.45, No. 1, pp. 357-370, ISSN 1359-6454
- David, D. et al (1979). Etude de la diffusion de l'oxygene dans le titane  $\alpha$  oxyde entre 700°C et 950°C, *J.Less-Comm. Met.*, Vol.65, No. 1, pp. 51-69, ISSN 0022-5088
- Fromm, E. and Gebhardt, E. (1976). *Gase und kohlenstoff in metallen*, Springer-Verlag, ISBN 3540072551, Berlin, Heidelberg, New York
- Grala, E. M. (1968). *Characterization of Alpha Segregation Defects in Ti-6Al-4V Alloy*, Technical Report AFML-TR-68-304, AD0845805, TRW, Inc. Cleveland, OH
- Le Claire, A.D. & Neumann, G. (1990). In: *Diffusion in Solid Metals and Alloys*, Mehrer H. (Ed.), Vol.III-26, pp. 85- 212, Landolt-Börnstein, Springer-Verlag, ISBN3-540-50886-4, Berlin

- Le Claire, A.D. (1990). In: *Diffusion in Solid Metals and Alloys*, Mehrer H. (Ed.), Vol.III-26, p. 471, Landolt-Börnstein, Springer-Verlag, ISBN3-540-50886-4, Berlin
- Mitchell, A. (1984). Final Report to General Electric Corp., pp. 10-29, University of British Columbia, Vancouver, Canada
- Reddy, R.G. (1990). Kinetics of TiN Dissolution in Ti Alloys. In: *Electron Beam Melting and Refining State of the Art*, R. Bakish (ed.), 119-127, NV: Bakish Materials, ISBN-10 9992384719, ISBN-13 978-9992384718, Reno
- Schwartz, F. (1993). Technical Report YKOG, No 3028/93, pp. 2-7, SNECMA, Paris, France
- Tarenkova, N.Yu. et al. (2011), Formation of Gas-Saturated Defects in Titanium Alloys during Vacuum-Arc Remelting, Russian Metallurgy (Metally), Vol.2011, No. 2, pp. 127-132, ISSN 0036-0295
- Vykhodets, V.B. et al. (1987). Oxygen diffusion in  $\alpha$ -Ti. I. Anisotropy of oxygen diffusion in  $\alpha$ -Ti, The Phys. Metal & Metallogr., Vol.64, pp. 127-133, ISSN 0031-918X
- Vykhodets, V.B. et al. (2006). Studying distribution of gaseous impurities and carbon in titanium alloys using nuclear microanalysis, The Physics of Metals and Metallography, 2006, V.101, No.3, pp. 267-275, ISSN 0031-918X
- Vykhodets, V.B. et al. (2007). Genesis of Metallurgical Defects in Titanium Alloys, Doklady Physical Chemistry, Vol.416, part 2, pp. 285-288, ISSN PRINT 0012-5016, ISSN ONLINE 1608-3121
- Vykhodets, V.B. et al (2011). Identification of the sources of gas containing metallurgical defects during the smelting of titanium alloys, Russian Journal of Nondestructive Testing, Vol.47, No. 3, pp. 176-188, ISSN 1061 8309
- Wood, F. W. (1969). *Elimination of Low-Density Inclusions in Titanium Alloy Ingots*, Final technical report 1 Sep 67-30 Aug 68, AD0852028

IntechOpen



## **Titanium Alloys - Towards Achieving Enhanced Properties for Diversified Applications**

Edited by Dr. A.K.M. Nurul Amin

ISBN 978-953-51-0354-7

Hard cover, 228 pages

**Publisher** InTech

**Published online** 16, March, 2012

**Published in print edition** March, 2012

The first section of the book includes the following topics: fusion-based additive manufacturing (AM) processes of titanium alloys and their numerical modelling, mechanism of  $\beta$ -case formation mechanism during investment casting of titanium, genesis of gas-containing defects in cast titanium products. Second section includes topics on behavior of the  $(\alpha + \beta)$  titanium alloys under extreme pressure and temperature conditions, hot and super plasticity of titanium  $(\alpha + \beta)$  alloys and some machinability aspects of titanium alloys in drilling. Finally, the third section includes topics on different surface treatment methods including nanotube-anodic layer formation on two phase titanium alloys in phosphoric acid for biomedical applications, chemico-thermal treatment of titanium alloys applying nitriding process for improving corrosion resistance of titanium alloys.

### **How to reference**

In order to correctly reference this scholarly work, feel free to copy and paste the following:

Vladimir Vykhodets, Tatiana Kurennykh and Nataliya Tarenkova (2012). Genesis of Gas Containing Defects in Cast Titanium Parts, Titanium Alloys - Towards Achieving Enhanced Properties for Diversified Applications, Dr. A.K.M. Nurul Amin (Ed.), ISBN: 978-953-51-0354-7, InTech, Available from:

<http://www.intechopen.com/books/titanium-alloys-towards-achieving-enhanced-properties-for-diversified-applications/genesis-of-gas-containing-defects-of-metallurgical-origin-in-titanium-alloys>

**INTECH**  
open science | open minds

### **InTech Europe**

University Campus STeP Ri  
Slavka Krautzeka 83/A  
51000 Rijeka, Croatia  
Phone: +385 (51) 770 447  
Fax: +385 (51) 686 166  
[www.intechopen.com](http://www.intechopen.com)

### **InTech China**

Unit 405, Office Block, Hotel Equatorial Shanghai  
No.65, Yan An Road (West), Shanghai, 200040, China  
中国上海市延安西路65号上海国际贵都大饭店办公楼405单元  
Phone: +86-21-62489820  
Fax: +86-21-62489821

© 2012 The Author(s). Licensee IntechOpen. This is an open access article distributed under the terms of the [Creative Commons Attribution 3.0 License](https://creativecommons.org/licenses/by/3.0/), which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

IntechOpen

IntechOpen