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Fractality of Fractures of Aluminum and Titanium Alloys Irradiated by Intensive Electron Beam

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The aluminum and titanium plates were irradiated by the high-current electron beam with the electron energy around 0.35 MeV, impulse duration of 5 μs, beam current of 2 kA, and with the incident energy density up to 3.5 MJ/m^2. The cross-fractures were made in the modified and non-irradiated areas. The fracture surfaces were examined using a SEM JEOL JSM-840. The irradiation resulted in significant changes of the microstructure parameters (i.e. grain size, damage character). The fractal dimension of the grayscale SEM images of the fracture surfaces were statistically analyzed using the arithmetic, geometric and divisor step methods with the sliding square window of varying size. The calculated distributions of fractal dimensions helped to characterize the scaling behavior of the microstructures, which accompany a shift of the fracture mechanism into preferably brittle mode.

Keywords: Fractal dimension, Electron beam, Aluminum, Titanium, Ablation.

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1. INTRODUCTION

The materials properties under exploitation in extreme conditions could be significantly changed due to the impact of many interrelated factors, such as temperature, stress fields, ionizing irradiation, corrosion environment, etc. Studying microstructure enables identification of the changes, what is necessary for further evaluation of technical parameters of materials. Microstructural details (e.g., cracks propagation, grain boundaries, voids, ridges), which are generally characterized by the complex geometrical form, define its physical, mechanical properties. The typical approach to study the microstructure of some material is analyzing its fracture surfaces. There is a great variety of different techniques and methods, which could be applied for, but this paper focuses only on the fractal analysis.

This paper covers the obtained results on the fractal character of fracture microstructures modified by the electron beam exposure. The goal of the presented research is to give an insight on the fractal character of the fracture microstructures of materials before and after being exposed to the intense heat and shock loads.

High-current electron beam (HCEB) exposure has been used as an intense heat and shock-wave source, which impact usually induces extreme temperature and stress fields, leads to severe surface and/or bulk ablation, provokes significant changes in the microstructure [1]. HCEB-sublimation, melting, evaporation, recrystallization processes lead to grain growth/refinement, brittle-ductile fracture transition, etc. These changes are hard to describe in terms of usual Euclidean geometry. The damage mechanisms, plasticity, evolution of defects, and even thermoconductance become more complicated due to the local fractal behavior of the microstructure.

The samples of the industrial Aluminum alloy 1933 of Al-Zn-Mg-Cu system, and technically pure Titanium alloy VT1-0 were selected for this study, which physical-mechanical parameters (e.g., hardness, chemical composition, damage behavior) and temperature dynamics previously were studied in [2, 3]. At present, it is of great interest to conduct research on fractality of SEM images of the fractures of the irradiated and 'as-fabricated' materials to find its links to the macroscopic parameters as well as to the applied energy impact.

Worth noting, the term 'fractality' means the statistical estimation of the fractal dimension. The real image sets are always continuous and have limited resolution, even if they were taken in the wide scale interval. Thus, it is impossible to employ the basic property of mathematically correct fractals – self-affinity, so, we concentrate on the statistical self-affinity of the limited discrete data, which represents the grayscale SEM images. It should be pointed out, that the values of such statistically fractal objects cannot be evaluated exactly, because of their above-mentioned nature. Moreover, the calculated values depend on the applied numerical method. So, we could only chose several appropriate methods to evaluate the approximate values.

In [4], it was recommended to choose the triangular prism methods (TPM) for analysis of the complex surfaces, because they are more reliable estimators than the isarithm, variogram or box-counting methods. Thereof, we used in our calculations the different modifications [5-7] of the basic TPSAM proposed by Clarke in 1986 [8].

The size of optimal moving window, which provides the accurate detection of fractal parameters of certain heterogeneities in data sets, could be closely associated with the specific sizes of these heterogeneities. It should be kept in mind, that fractality does not show

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the pixel uniformity for large pictures; such uniformity may be detected if its size does not exceed the window size. However, real image usually has a complex multifractal character impossible to determine using only a few window sizes.

The next aspect to mention is the source of fractality. It is assumed, that the fractal properties of SEM images are caused by the intensity variation, which could be characterized by the fractional dimension D_{frac}. However, an existing noise during detecting along with the hardware-induced distortions could also be characterized by the fractal nature. Another point which has to be taken into consideration is how does the sliding ('moving') windows of certain sizes distort the initial data during the calculation processes, and how to discriminate these distortions from the final useful data.

In spite of faced problems, which cannot be answered in this paper and what makes fractal analysis of SEM images more complicated, we present our investigation on fractal nature of the fractal nature of SEM images, which could be of further help for developing the crystal plasticity models, predicting damage evolution of materials under extreme condition.

The aim of our research is to investigate the fractal character of the SEM images of HCEB-irradiated Ti and Al in terms of statistical evaluation of fractal dimensions.

2. MATERIALS AND METHODS

The experimental samples were the plates of the aluminum alloy 1933 (Al > 90 %, 6.35 % Zn; 1.6 % Mg; 1 % Cu; 0.1 % Mn; 0.2 % Fe; 0.1 % Si; 0.06 % Ti; 0.05 % Cr; 0.1 % Zr, wt. %), and titanium alloy VT1-0 (Ti > 99.2 wt. %) [2, 3]. The initial square plates, which were subjected to HCEB-exposure, had the dimensions of 90 mm × 2 mm (w, $l \times t$) for 1933 alloy, 100 mm × 2 mm (w, $l \times t$) for titanium.

The prepared specimens were irradiated at the TEMP-A pulsed electron beam accelerator facility in the Institute of Plasma Electronics of the NSC KIPT NAS of Ukraine. The general parameters of HCEB exposure were: the quasirelativistic electron energy around 0.35 MeV, incident beam current ~ 2 kA, impulse duration $\tau_{imp} \sim$ 5 μs, beam's cross-section was hollow with the Gaussian-shaped wall, pressure in the vacuum camera did not exceed 10^{-5} torr, peak intensity of irradiation was up to 10^{12} W/m^2. The samples were properly fixed to the collectors. Necessary to mention, that the sample-beam interactions were not affected significantly by the collateral phenomena of the beam-holder interactions in any case (e.g., deposition of the melted material of holder onto the target's surface was negligible due to shielding by the expanding dense plasma cloud from the target's surface).

After HCEB irradiation, the surfaces of all samples were examined using the optical microscope Bresser BioLux NV. Then, the perpendicular fractures were made in the epicenter and periphery zones at the temperature around 280 K. The fracture processes were not enough accurate due to small sizes of samples and absence of cryogenic cooling, so, their fracture morphology became more complicated to analyze because of severe deformation. SEM fractography was carried out using JEOL JSM-840 microscope at the V.N. Karazin Kharkiv National University.

To reveal the fractal parameters of the microstructures before and after exposure, the fractal dimension D_{frac} distributions of the grayscale SEM images of the fractures were built. SEM image can be represented as a 3D matrix, which describes the intensity variation over a projection area of a detector. This means, that we operate only with a degenerated algebraic 3D surface, which usually does not thoroughly specify the real fractal measures of the examined surface, and seriously distort its fractal descriptors. Nevertheless, it is assumed, that the obtained SEM data could provide some general data about the surface fractality.

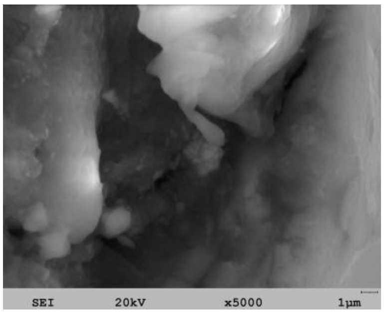
D_{frac} distributions were statistically calculated using the modifications of the classic Clarke's triangular prism surface area method (TPSAM) [8]. The applied numerical procedures could be classified by the calculation methods for its constituent subroutines in the following way: (i) by the method of calculation of the surface area of a prism: classic Clarke's 1986 TPSAM [8], and eight-pixel, max-difference, mean-difference introduced by Sun in 2006 [5]; (ii) by the method of plotting the log-log regression line (regression of log of area vs. log of step size): the squared step size method (original Clarke's TPSAM) and the linear step size method proposed by Qiu in 1999 [4]; (iii) by the step size increment of the grid in the window: geometric, arithmetic, divisor-step [4-8], and (iv) by the size and move direction of the window: fixed, automatic, and random, gliding, respectively. Each numerical procedure comprises certain calculation subroutines of (i), (ii), (iii) and (iv). The great variety of the numerical methods enables determining the optimal way to detect and recognize special inhomogeneity in data, which has apparent deviation from Euclidean geometry. In this work, the geometric-square windows with variable side length were employed over the data sets with the unary moving step for calculation of the local D_{frac} using the mentioned above subroutines.

The mentioned numerical algorithms as well as supplementary decoding and visualization were implemented in Pascal/Delphi. The calculation procedures were validated using the tests available in the early work of Clarke [8]. The codes, compiled executive files for Win7 and additional test examples could be found in GitHub via link [9].

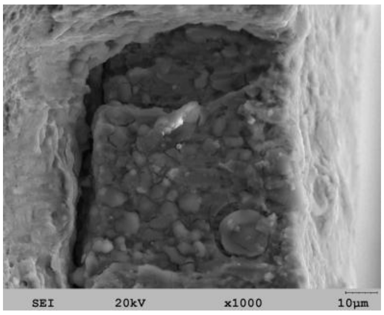
3. RESULTS

The irradiation of metals by the quasirelativistic HCEB can be simply compared to welding, regarding the deep modification of the targets. However, the time of HCEB exposure is very short, its intensity and energy are much higher than in the case of welding, which lead to the thermal explosion of the surface layers, with propagation of shock-waves into the bulk of material [1]. In general, the phenomena occurred during HCEB irradiation, are very complicated even for homogeneous targets.

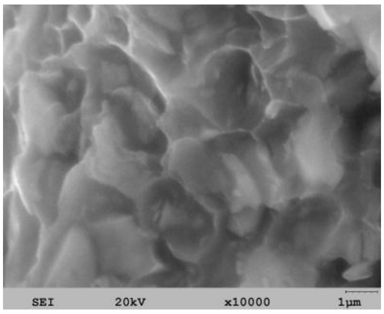
The final microstructures of aluminum and titanium after irradiation have typically quenched character in the upper surface layer (Fig. 1a, 2a) [2, 3], then it changes to the melted-and-heat-affected zone deeper into the bulk (Fig. 1b, 2b), which finally disappears in the non-modified material (Fig: 1c, 2c).



a



b

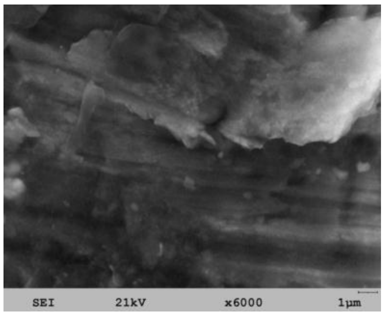


c

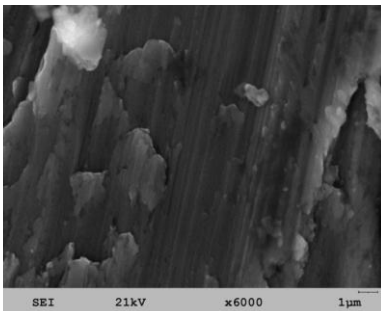
Fig. 1 – Fractures of aluminum: (a) – quenched layer, (b) – melted and heat-affected layer, and (c) – non-irradiated material in the bulk of the sample

We assume, that the variation of intensity of SEM image is a collective characteristic, which depends on the fracture roughness, grain shape and curvature, while the beam current is steady, as well as brightness and focus are optimal for each scan. For the sake of clear understanding, the calculated fractal dimensions do depend very much on the expertise of the microscope operator (i.e. how does a SEM image is taken: angle, contrast, magnification, stigmatism correction, etc). But to simplify the problem, we neglect these parameters. Thus, fractal analysis in such a case can be applied for the SEM images to describe qualitatively the microstructures.

Having tested the different forms of a window, it was decided to employ the right square form of window to minimize other possible distortions in the D_{frac}-maps besides the cross-like blurring. Fractal analysis methods were tested using all mentioned constituent subroutines. Some methods (e.g., original Clarke's TPSAM [8]) slightly underestimate Dfrac, other methods usually overestimate it (e.g., geometrical method with linear step size gives 20..30 % higher results). We found the Clarke's method with the arithmetic step size within a window with size of 9 px × 9 px, and squared step size in log-log regression, to be the most robust method for analysis in a wide interval of fractional dimensions from a vicinity of 2 up to 3.



a



b

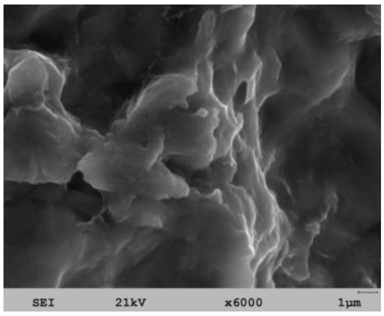


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Fig. 2 – Fractures of titanium: (a) – quenched layer, (b) – melted and heat-affected layer, and (c) – non-irradiated material in the bulk of the sample

It was noticed, the unmodified aluminum alloy 1933 has the compacted structure of brittle facets within a size interval of 0.6..1.5 μm (see Fig. 1c). Its quenched zone has the coarse-grained structure, which consists of disoriented grains with linear dimensions in rage of 1..3 μm and its fracture is predominantly brittle (Fig. 1a). Melted zone is characterized by the micro-brittle fracture mechanism of equaixed grains with a linear size 1..2 μm, with many intercrystalline cracks (Fig. 1b).

The initial microstructure of VT1-0 alloy has the complexed fracture surface with high roughness. It is a typical example of a ductile damage. The non-irradiated material has the equiaxed globular grains with linear size not more than 10 μm. The quenched and heat-affected-zone can be characterized by the brittle fracture and by the significantly bigger grains up to 100 μm. It has a lamellar structure of elongated grains parallel to the surface. Titanium in this melted layer has small amount of intergrain cracks.

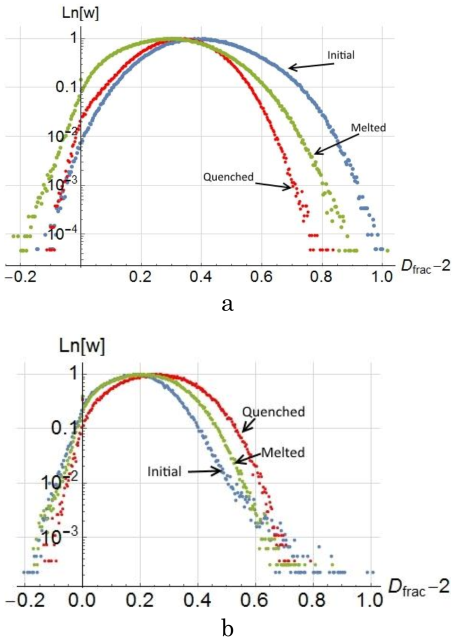

Fig. 3 – Logarithmic normalized probability distributions w of D_{frac} of: (a) aluminum alloy 1933 and (b) titanium alloy VT1-0 in the unmodified, quenched and melted zones

The mentioned above embrittlement in both cases is reflected in the D_{frac} spectra (Fig. 3). On practice, the peaks of calculated distributions are in similar position, but their left and right tails together with tilt are different. The left tail goes to binary dimensionality, which describes the case, when all the points of a data set have the same value. The right tail of the distribution and tilt provide us most of information about fractality and randomness. For aluminum (Fig. 3a), the right tail of D_{frac} distribution decreased by 10..20 % as a physical reduction of the surface microcomplexity. In contrary to aluminum, the behavior of D_{frac} for titanium is opposite in general case (see Fig. 3b). Its value increased for the quenched and melted zones. The transformation from ductile into brittle accompanies with the complication of the local microtopology by the surface ripples, fatigue striations and grain growth. The surface ripples are considered to be responsible for such an increase of fractal dimension for irradiated titanium. To prove this hypothesis, we changed the brightness and contrast of images of this area in order not to see the ripples, and then binarized them to distinguish the basic features such as shapes of grains, fracture cavities. This procedure was done for all characteristic zones for comparison. The calculated fractal behavior after selective filtering and binarization was similar to the aluminum fractures: Dfrac decreases by 10..20 % after quasirelativistic HCEB treatment. The only difference of Dfrac for the 'as-taken' images and filtered images, is in the grain size; shape and local details in fine scale, which highlights the importance of scaling in statistical evaluation of D_{frac}.

The concept of fractal nature of SEM images of fractures is still complicated and ambiguous issue, and requires a comprehensive exploration.

4. CONCLUSIONS

In this paper, the physical nature of the cross-fractures of aluminum alloy 1933 and titanium alloy VT1-0 after the high-current electron beam exposures has been studied based on the fractal dimension characteristics. The e-beam irradiation resulted in a significant modification of the collective fracture behavior of the materials, which was examined by SEM imaging. It was found, that fractal dimension D_{frac} in the modified zone of aluminum decreased by 10..20 % as a result of reduction of local complexity. In the case of titanium, D_{frac} slightly increased due to appearance of ripples and striations, compared to the unmodified area. However, the average fractality of fractures, except the mentioned striations, decreased in the same way as for aluminum. The obtained data indicate the correlation of fractality with the changes of topological fracture behavior from the ductile in the unmodified area, to brittle in the quenched, heat-affected zone, which reflects the character of the microdamage mechanisms.

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Фрактальність поверхонь зламів алюмінію і титану опромінених інтенсивним електронним пучком

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Опромінено алюмінієву і титанову пластини сильнострумовим електронним пучком з енергією електронів 0.35 МеВ, тривалістю імпульсу 5 мкс, струмом пучка 2 кА, з густиною енергії до 3.5 МДж/м². Поперечні злами зроблено в зонах немодифікованого і опроміненого матеріалу, які було проаналізовано за домомогою скануючого електронного мікроскопу JEOL JSM-840. Опромінення призвело до суттєвих змін параметрів мікроструктури (і.е. розміру зерна, характеру руйнування). Фрактальну розмірність зображень мікроскопії зламів в сірих відтінках статистично обчислено використовуючи арифметичний, геометричний, поділу кроку методи з ковзаючим квадратним вікном змінних розмірів. Отримані розподіли фрактальної розмірності допомогли охарактеризувати скейлінговий характер мікроструктур, що супроводжує зсув механізму зламів в преференційний крихкий режим.

Ключові слова: Фрактальна розмірність, Електронний пучок, Алюміній, Титан, Абляція.

Фрактальность поверхностей изломов алюминия и титана облученных интенсивным электронным пучком

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Облучено алюминиеву и титановую пластины сильноточным электронным пучком с энергией электронов 0.35 МэВ, длительностью импульса 5 мкс, током пучка 2 кА, плотностью энергии до 3.5 МДж/м^2. Произведены поперечные изломы в зонах немодифицированного и облученного материала, которые проанализированы с помощью сканирующего электронного микроскопа JEOL JSM-840. Облучение привело к существенным изменениям параметров микроструктуры (i.e. размера зерна, характера разрушения). Фрактальную размерность изображений микроскопии изломов в серых оттенках статистически рассчитано используя арифметический, геометрический, деления шага методы с скользящим квадратным окном изменяемых размеров. Полученные распределения фрактальной размерности помогли охарактеризовать масштабный характер микроструктур, что сопровождается сдвигом механизма изломов в преимущественно хрупкий режим.

Ключевые слова: Фрактальная размерность, Электронный пучок, Алюминий, Титан, Абляция.

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