

Niobium cavitation analysis by SEM

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Abstract. Cavitation erosion is common in hydraulic turbines and presents a costly trouble for this power generation utilities. Different materials have been studied for the turbines blades fabrication and recovering. In this work, the cavitation behavior of pure niobium was analyzed. The cavitation experiment was made in a standard vibratory cavitation erosion test as a function of test time. The time tests of niobium samples were 10, 30 and 50 min. The results are compared to AISI 1020 cavitation behavior. The niobium samples were characterized by Scanning Electron Microscopy (SEM). The wear test showed that the pure niobium had a poor cavitation wear resistance.

Keywords: Niobium, Cavitation

1. Introduction

The history of niobium (also called columbium in American English) is tightly connected with that of tantalum. Both metals share very similar chemical characteristics and are always found together in nature. In the year 1801, niobium was discovered by the English Chemist Hatchett. Now, the common name for this metal is niobium. It was coined by its second discovering (H. Rose). The semi finished products made of niobium and niobium alloys (e.g. Nb1Zr) are manufactured by means of sintering as well as melting. This material presents some characteristics such as; gray or silvery, ductile metal and it doesn't tarnish or oxidize at room temperature. It reacts with oxygen and halogens only when heated. It is less corrosion-resistant than tantalum at high temperature. The niobium does not attack by nitric acid up to 100 °C, but it attacks vigorously by mixture of nitric and hydrofluoric acids. Hot, concentrated hydrochloric, sulfuric, and phosphoric acids attack it, but hot, concentrated nitric acid does not. In the room temperature it is unaffected by most acids and by aqua regia. It is attacked by alkaline solutions to some extent at all temperatures [1-2].

Niobium is also used as an additive in super alloys for jets and turbine engines and as carbide in machining cutting tools. Its volume in nuclear, aerospace, and superconducting applications is increasing each year. As a metal niobium was first used in the atomic reactor program. Niobium is relatively light in weight and can maintain its strength at elevated temperatures, because of it is used extensively in aerospace equipment and missiles. Since niobium exhibits excellent corrosion resistance to liquid metal it is used for parts in sodium vapor and magnesium lamps for highway lighting. In alloyed condition with titanium or tin the niobium has become the primary material used in superconducting applications [3]. Studies of niobium addition in hot rolled steel condition had also been done. These steels with near eutectoid composition presented maximum variation in yield and fatigue limits with smallest additions of niobium metal [4].

The cavitation phenomenon is characterized by vapour generation and condensation. It occurs frequently in hydraulic machines. It causes vibration, increase hydrodynamic drag, change in the hydrodynamics flow, noise, thermal and light effects like luminescence and the most important of all is cavitation erosion. Rayleigh was the first to study the cavitation erosion problem on the ship propellers [5]. Since then various studies have been conducted in this area, comprising studies in weldments, stainless steel and coatings. Their objectives have been simulating the real damage in surface as function of time and know the better material to use in those conditions.

The studies about cobalt-alloyed stainless steel cavitation the removal material initiates and propagates from slip steps and grain boundaries. The advantage stage of erosion involves a predominantly ductile fracture mechanism. The long incubation period and low erosion rates of the alloy can be related to the high work hardening rate and high fatigue resistance [6].

A refined microstructure is also a requisite for a good performance against cavitation, as the fine grain size produces smaller eroded particles and consequently results in a lower erosion rate in cobalt-alloyed stainless steel cavitation. The higher resistance against cavitation associated to the finer grain size would be explained by an increase in the work necessary to remove the material, related to the expression from Hall-Petch [7]. Studies showed that the sanded sample condition presented inferior resistance to cavitation. The risks originated in sanding generate nucleation of erosion process [8].

This work studied the cavitation behavior of pure niobium. The cavitation experiment was made in a standard vibratory cavitation erosion test as a function of test time. The results were compared to AISI 1020 cavitation behavior. This work also

studied surface aspects of the cavitation eroded material related to the morphology and to the failure mechanism, which were analyzed by SEM.

2. Experimental and materials

The niobium samples of 20x35x5mm were laser cut from a niobium cold-rolled bar. Samples were sanded 400, 600, 1200 and 2000 granulometry and polished using Al_2O_3 -water solution in agreement with the steel samples conventional metallographic procedure.

The cavitation-erosion experiments were carried out by using a vibratory apparatus according to ASTM G 32 [9]. The test, as the ASTM G 32, basically consists of measuring the weight or volume loss of test specimen affixed to the tip of an ultrasonic horn that pulsates in a distilled water bath. In the present tests a modified procedure, illustrated in Fig. 1, was used, which facilitates the sample preparation. The AISI 1020 sample was cut from a hot-rolled bar. Niobium and AISI 1020 samples were submitted to the same experimental procedures.

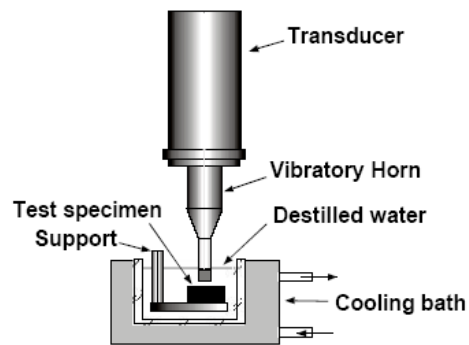


Figure 1. Modified cavitation test. After Boccanera [7].

The samples were cleaned for 6 minutes in an alcohol bath by ultrasonic and they were cavitated in Ultrasonic vibratory equipment with indirect method. The frequency applied was $20 \text{ kHz} \pm 0.2\text{Hz}$ and with amplitude of $50 \mu\text{m}$. The pointer was machined in AISI 304 stainless steel and polished until $1 \mu\text{m}$ superficial roughness.

The temperature of the distilled water bath was maintained at $21^\circ\text{C} \pm 1^\circ\text{C}$. The test specimens were placed in a distance of 0,5mm to the vibrating steel disc immersed 10 mm in distilled water. Specimen weight loss was measured up after each experiment. Measures were done in electronic scales with 0,1mg of precision.

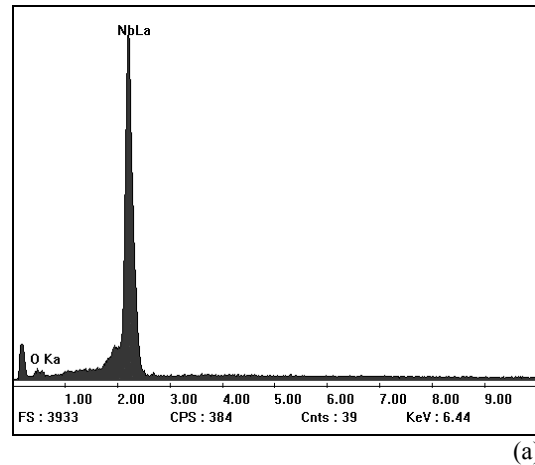
The niobium samples were submitted to different time of accelerated cavitations tests of 10, 30 and 50 min, respectively, and their weights were determinate before and after the tests.

They were characterized by Scanning Electron Microscopy (SEM) using Philips XL-30 equipment. The chemical composition of the niobium sample was obtained using energy dispersive X-ray analysis (EDX) and the microstructural analyses were done in magnitudes of 100x, 1000x and 5000x.

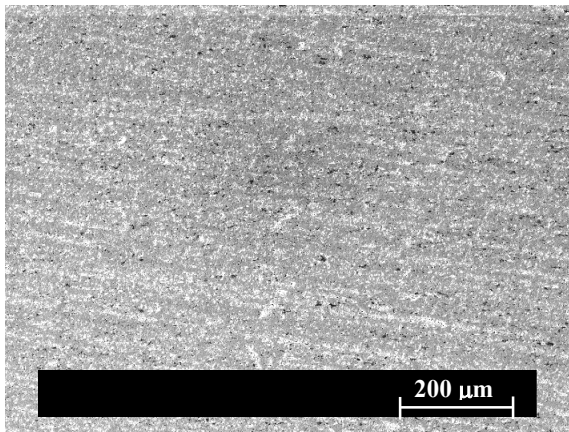
3. Results and Discussions

Figure 2 shows the result of non-cavitated niobium sample characterization that was analyzed by EDX and SEM comprising the chemical characteristics (Fig. 2a) and the surface aspects metal after polishing (Fig. 2b and 2c). As expected the EDX results confirm the high purity of the niobium samples.

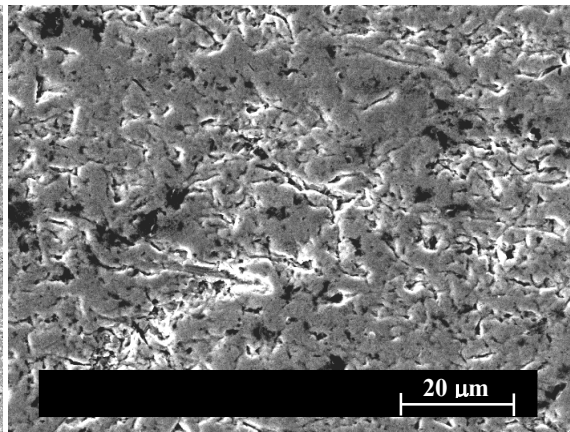
Figure 2(c) shows the utilized metallographic technique to polish the niobium sample results in a poor surface finishing. It is evidenced in the roughness surface due to the material removal resulting in the presence of a porous surface. The presence of small pores (black areas) in niobium surface samples is directly related with the polish method utilized, which was not adequate to niobium samples. Evidently, it may be concluded that the usual polish method to steel sample is not appropriated to niobium polish. It is necessary to make improvements to niobium polish procedure.



(a)



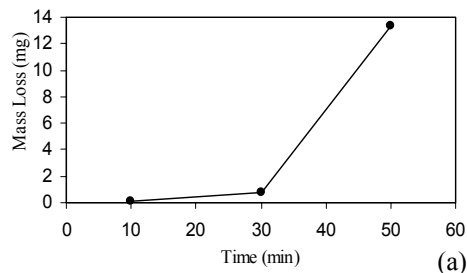
(b)



(c)

Figure 2. a) Aspect of non-cavitated niobium sample analyzed by SEM; b) Niobium sample surface aspect after polishing. Magnitude: 100x; c) Detail of (b) in magnitude of 1000x.

The materials losses were determined and the results are presented in Fig. 3. Figure 3(a) shows the evolution of the mass loss of the niobium samples as a function of the cavitation-erosion experiment time. As expected, the highest is the experiment time, the highest is the mass loss. This result is better observed in the table presented in Fig. 3(b), which indicates the quantitative values of the weight and percents mass loss.



(a)

Table 1. Samples loss weight as a function of the test time.

Time(min)	Δ mass (g)	Δ mass (%)
10	$0,1 \times 10^{-3}$	$4,10 \times 10^{-4}$
30	$0,8 \times 10^{-3}$	$13,3 \times 10^{-3}$
50	$13,3 \times 10^{-3}$	$5,40 \times 10^{-2}$

(b)

Figure 3. (a) Graphic of mass loss x time in niobium samples. (b) Mass loss of niobium samples tested.

On the other hand, it is useful to estimate the time required to attain maximum rate of erosion with knowledge of incubation period so that the total life of a particular component can be predicted. In accordance with Rao [10], which studied the role of incubation period in correlating and predicting the period of time required to attain the maximum rate of erosion, the erosion-time curve can exhibit four different zones, namely: 1) incubation period with no measurable weight loss; 2) accumulation zone with increasing loss-rate; 3) steady-state zone with approximately constant loss-rate; and 4) attenuation zone with decreasing

loss-rate. The first and third zones, ie, incubation and steady-state zones are of considerable importance to understand the basic mechanism of erosion and to evaluate the materials for erosion resistance and the total life of a material of a machine component. The incubation period can be defined in the followings two ways: (a) no measurable weight loss period, and, (b) intercept on time axis obtained by extending the portion of erosion-time curve. The period defined by (a) is generally termed as the incubation period, while the period defined by (b) is known as the initiation period. The existence of no weight loss period generally indicates that the failure of materials is caused by fatigue. On the other hand, the absence of this period strengthens the concept of single event pitting.

Figure 4(a) presents the definition of incubation period t_c , initiation period t_i , and the period of time t_m required to attain maximum rate of corrosion and fig 4(b) shows the mass loss curve of niobium samples cavitaded measures.

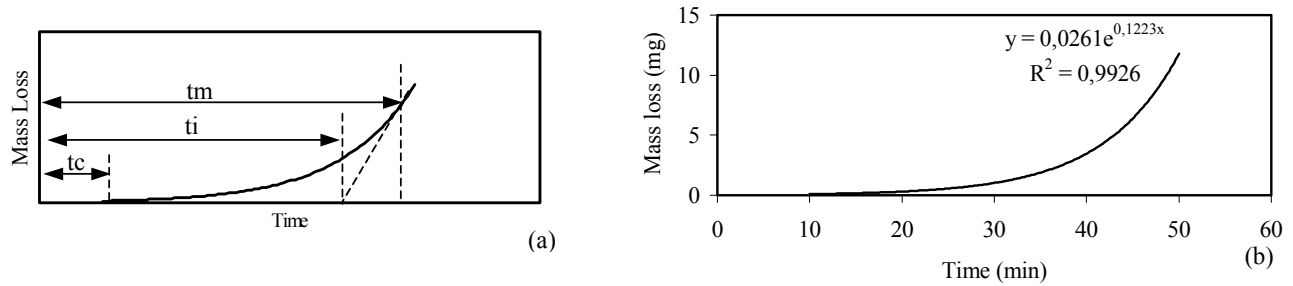


Fig. 4. (a) The relation of the incubation period, the initiation period and the period of time required to attain maximum rate of erosion (after Rao [10]). (b) Mass loss curve for niobium samples cavitaded measures.

The Figure 4(b) presents the curve obtained with the niobium samples cavitaded as function of test time. In agreement with Fig. 4(a) it is possible to predict the incubation period that was slightly lower than 10 minutes (another work is being performed for the determination of the niobium incubation period actual value).

In the Rao's work [10], the correlations of initiation period t_i and period of time to attain maximum erosion rate t_m with incubation period t_c are presented and it is point out that it is possible to predict t_i and t_m values and hence the total life of a component of a device with the knowledge of t_c . For the prediction of t_i and t_m values in field devices with flow conditions similar to those in venturi and rotating disc, Rao [10] presents the following simple generalized equations (1) and (2):

$$t_i = 2,9 * t_c^{1,15} \quad (1)$$

$$t_m = 4,0 * t_c^{1,15} \quad (2)$$

Then, applying Eq. (1) and (2) for the niobium results, ie, 10 minutes incubation period, the initiation period (t_i), for niobium sample, is 41 minutes and the period to attain maximum erosion rate (t_m) is 56 minutes. The calculated values are in agreement with the graphic procedure for the t_i and t_m determination, presented in Fig. 4(b).

Figure 5 shows a comparative of mass loss material between AISI 1020 and niobium samples. The cavitation-erosion

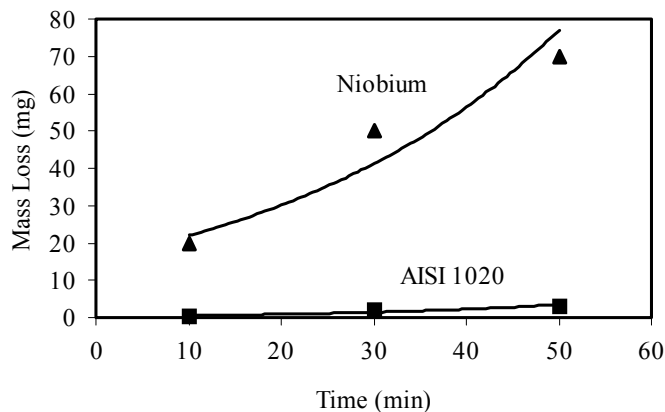


Figure 5. Comparative curves of cavitation experiments mass loss between AISI 1020 and niobium samples.

behavior for the both materials is very different. The AISI 1020 steel samples showed better cavitation-erosion resistance than niobium samples. It must be stated that pure niobium samples, in the condition that was studied, present a very poor cavitation-erosion resistance.

Figure 6 shows comparative electromicrographs of the niobium sample surfaces that were tested for 10, 30, and 50 minutes

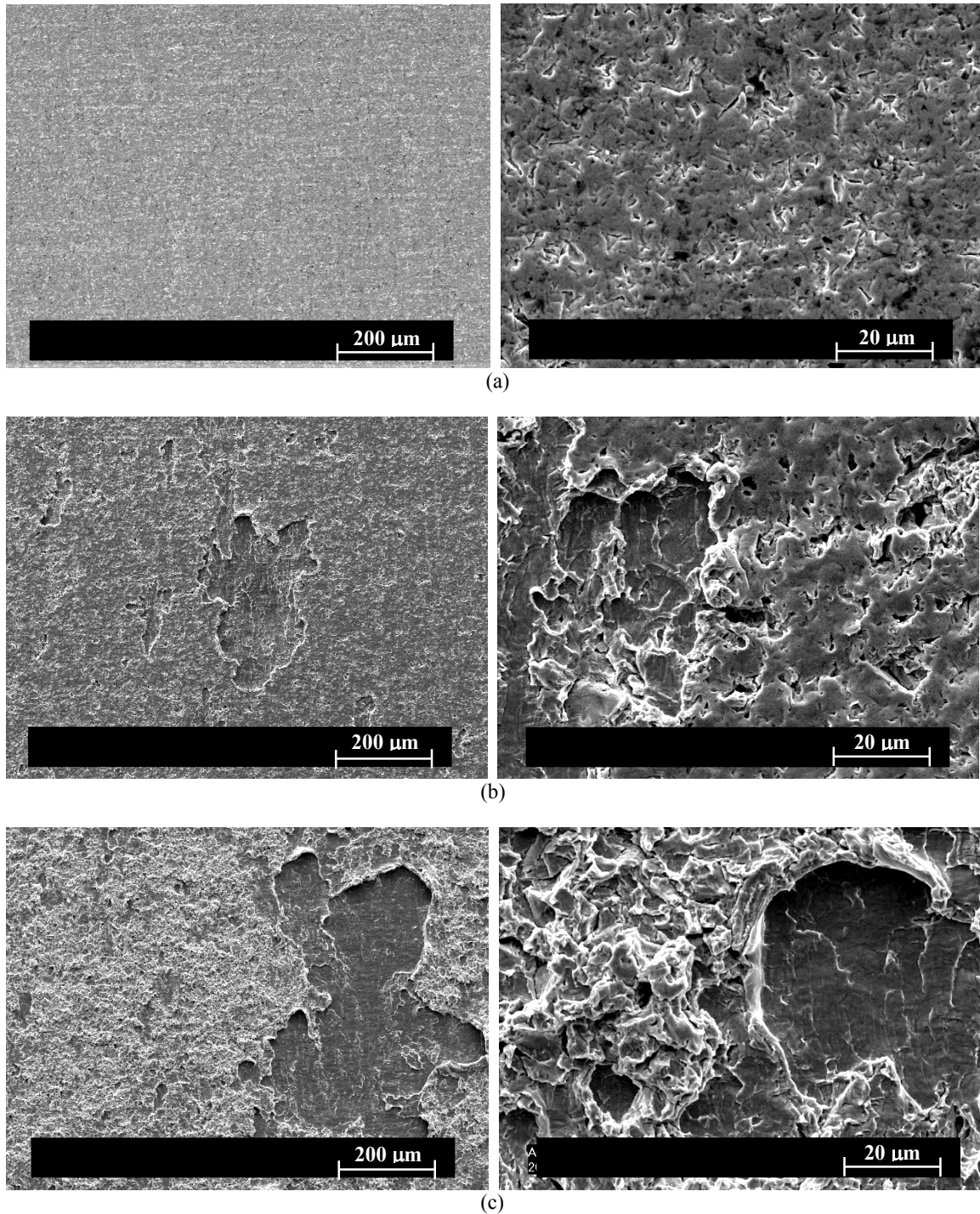


Figure 6. Aspects of the niobium sample surfaces cavitated for: a) 10 min; b) 30 min; and c) 50 min, in magnitudes of 100x, and of 1000x, respectively.

in magnitudes of 100x and 1000x. It is possible to compare the surface aspects related to the cavitation process. Obviously, the sample which test time is the highest its damage process is much more intense than the others. The cavitation-erosion process is better evidenced in Fig. 6(c) where depth extract of surface material occurs.

Figure 7 presents the surface aspects of non-tested and 10, 30 and 50 minutes tested niobium samples. In Figures 7(b, c, d) the aspect of the typical fatigue marks can clearly be observed (as indicated). These ridges are directly related to fatigue failure mechanism, which comprises the Rao [10] suspect, so the cavitation-erosion process is initiated by high number of cycles with no weight loss period. This also confirms that the failure of niobium material is caused by fatigue.

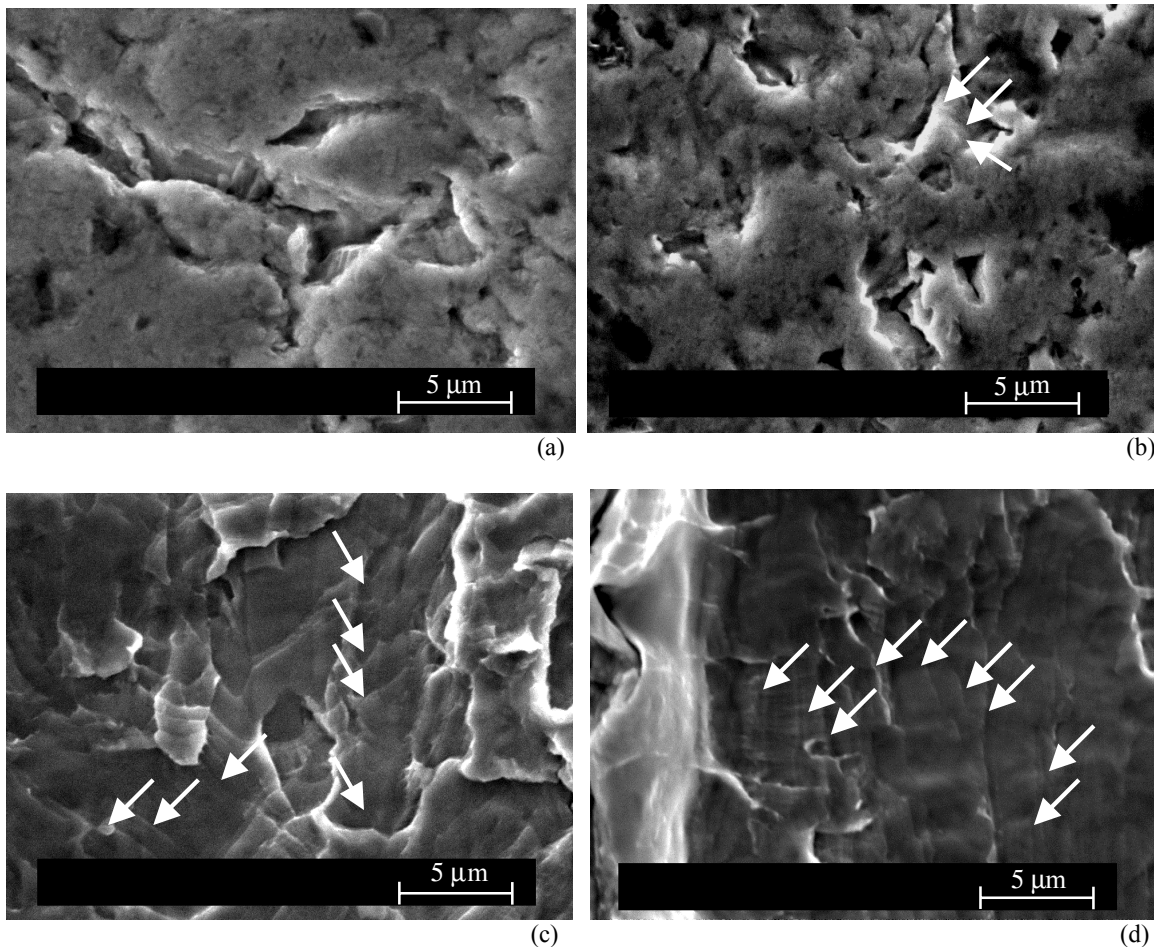


Figure 7. Aspects of the niobium sample surfaces all in magnitude of 5000x: (a) Sample non-cavitated. (b) 10 min cavitated. (c) 30 min cavitated. (d) 50 min cavitated.

4. Conclusions

This work studied the cavitation behavior of pure niobium. It was evidenced by the SEM analyses the failure of the cavitated niobium samples is caused by fatigue. Besides, it was concluded the pure niobium presents a poor cavitation-erosion resistance. The fact that the niobium surface was prepared utilizing the conventional metallographic procedure could be a favourable factor to cavitation process. So, another work is being performed to determine the actual influence of the surface initial condition on the cavitation-erosion process in niobium.

5. References

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