Early stage cavitation erosion within ceramics – An experimental investigation

G. García-Atance Fatjó, M. Hadfield, C. Vieillard, G. Morales.

Abstract

Four material types were considered within an experimental investigation to identify the failure mechanism resulting from cavitation exposure. These materials were zirconia, silicon nitride and alumina with stainless steel as reference. An ultrasonic transducer was utilised to produce cavitation conditions and the configuration was "static specimen method" using a 5mm diameter probe, 20kHz and 50μm of amplitude. The exposure times were periods from 15 seconds to 2 hours.

Experimental methods employed to characterise wear mechanisms were light microscopy, scanning light interferometry, scanning electronic microscopy.

It was found that zirconia and silicon nitride demonstrated evidence of plastic deformation. Zirconia showed evidence of time delayed for transformation of phase. Alumina showed evidence of fracture type failure mechanism with negligible plastic deformation. All wear mechanisms are discussed and the materials are ranked in terms of cavitation resistance performance.

1. Introduction

Cavitation erosion behaviour of ceramics has been studied traditionally from the point of view of material loss rate and incubation time [1] [2] [3]. Incubation time has been measured as the time when a weight loss of 0.1mg is detected [1]. This approach is suitable in application where the form keeping is the critical factor.

There are some applications where ceramics are used in such condition that keeping the roughness is a key factor and where cavitation can be a determinant factor to produce erosion. An example of this application is ceramic bearings working in low saturation lubricants. Not too much work has been made studying the materials behaviour in the early stages of cavitation erosion in ceramics. C. Haosheng et al. study the damages on steel surface at the incubation stage of cavitation erosion. They show the mechanisms of pit formation in this stage [4] [5].

On the other hand, there have been studies focussing the cavitation itself [6]. Moussatov et al. has studied about the cone-like bubble formation that occurs when there is no stationary specimen in front of the probe [7] and ultrasonic cavitation in thin liquid layers [8], that is important in this study due to its relation with the configuration of the test that has been used. Moussatov shows that there is an amplification factor in the cavitation produced in the central point of a cylindrical thin layer by means of an ultrasonic device.

In our ongoing study, comparison of ceramic material and stainless steel cavitation erosion behaviour in early stages has been carried out. The initial plastic deformation due to the shock pressure plays an important role in the mechanism of damage. A ranking of the materials performances has been made.

2. Testing methodology

The standard ASTM G32-03 [9] gives the method to test cavitation erosion in brittle materials. The standard names it as "stationary specimen method". This test consists in exposing the material close to the face vibratory tip of the probe. Fig. 1. In this configuration the geometry characteristics of the specimen are very important because variations of these change the cavitation intensity due to geometrical amplification factors [8]. The gap between the probe and the sample is 0.5mm and the diameter of the probe is 5mm, the specimens are flat. Table 1. shows the roughness of the samples before tests, all the samples have been

polished following the same method, with diamond slurry. A feeler gauge is used to make sure the gap is 0.5mm.

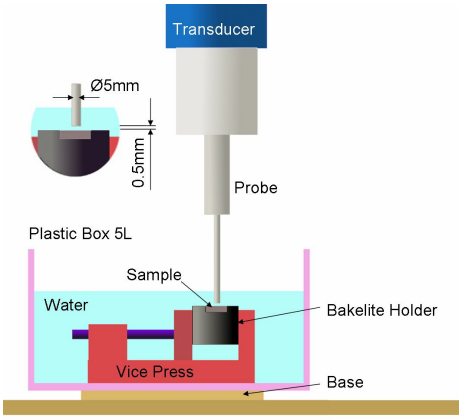

Fig. 1. Test assembly

	R_a	*rms*
Alumina	0.019µm	0.060µm
Zirconia	0.003µm	0.004µm
Silicon Nitride "A"	0.001µm	0.002µm
Silicon Nitride "B"	0.003µm	0.003µm
Silicon Nitride "C"	0.001µm	0.003µm
Silicon Nitride "D"	0.008µm	0.010µm
Stainless Steel	0.001µm	0.001µm
Definitions	$R_a = \frac{1}{L}\int_0^L \lvert z(x)\rvert dx$	$rms = \sqrt{\frac{1}{L}\int_0^L z^2(x)dx}$

Table 1. Roughness of samples before tests.

An ultrasonic piezoelectric transducer has been used, this transducer oscillates in the frequency of 20kHz and with an amplitude of 8.9µm. Whereas the titanium probe has a natural resonance of 20kHz and a magnification amplitude of 5.6 measured with the optical microscope. Then, the oscillation amplitude of the probe tip is 50µm. The electric working power of the system was approximately 7 Wats with this configuration and this gap. The ASTM Standard [9] recommends the values of 20kHz and 50µm. The water temperature was the room temperature 22±2ºC and the depth of the sample was 10±2mm.

Optical microscope photos with polarizing filter and without polarizing filter are taken with x5, x10, x20, x50, x100 magnification objectives at the next exposure times: 0sec, 15sec, 30sec, 45sec, 1min, 1.5min, 2min, 2.5min, 3min, 4min, 5min, 6min, 8min, 10min, 12min, 15min, 20min, 25min, 40min, 60min, 90min, 120min. These photos are always taken of the same place in the sample. This place is coincident with the point bellow the central point of the probe tip in the assembly. To make sure this, an acetate sheet with the position of the probe and with the central targeting point of the microscope is used. Besides the central point marked on the acetate, several characteristic reference on the surfaces of the samples are used to locate with more precision the samples in the microscope. This references were close to the border of the visual image within the ocular lens. Whenever there was not a characteristic on the surface suitable to use as reference some metal debris was deposited rubbing with a sharpened metal tip. This debris is small enough and is far enough from the centre of the cavitation erosion to not vary the results.

With this process, there is a record of the surface along the cavitation erosion in early states. The photos with polarizing filter are useful to note the small initial damage on the surface, because any small change in the slope of the surface is visible with the filter and

because plastic deformation will create a slope. The photos without polarizing filter are useful to note the cracks and material loss, because, in this case, slopes are not visible, and these characteristics are the only ones that typically are visible.

A Profiler Interferometer has been used to measure the damage on the surface of the samples. Measurements have been taken at 0sec, 60min, 120min. The measurement surface was always the centre of the cavitation damage. It has been used, as well, to measured the plastic deformation pits in the first minutes of exposure to cavitation.

SEM photos have been taken in order to have a better understanding of the erosion mechanisms, this was specially useful when a big plastic deformation occurred. The photos have been taken after 3 hours of cavitation exposure.

The ranking of cavitation erosion performance of material has been made according to the following criteria: plastic deformation pit volume, surface roughness, number of plastic deformation pits and surface loss.

Finally, photos of a water drop on the surface of the sample have been taken in order to check if different hydrophobic characteristics of the surface affect the cavitation erosion performance. In a hydrophobic surface is more likely the existence of nuclei where the cavity starts to grow and can collapse close to the surface [10]. The influence of this factor has been rejected as no important due to the similarity of hydrophobic characteristic of the surfaces.

3. Analysis of results.

3.1 Silicon nitride.

Silicon nitride from different manufactures has been tested. The fig. 2 shows the damage produced in the silicon nitride "A" in the first 15 seconds of cavitation exposure. This damage is small plastic deformation pits. The pits that are not targeted with a small arrow come from the finishing polish.

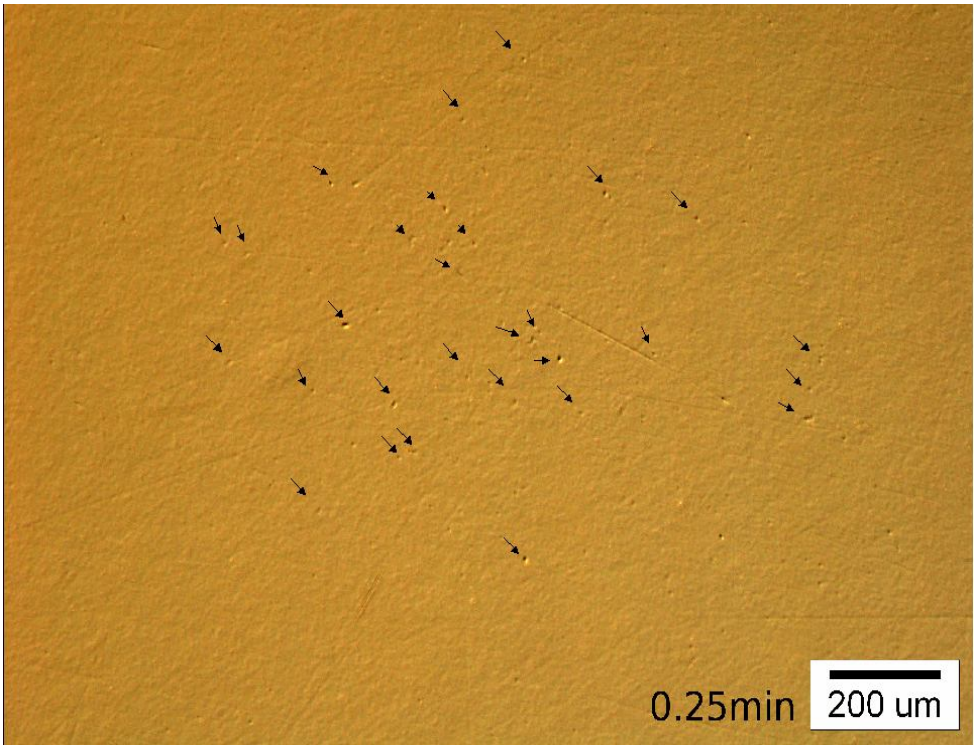

Fig. 2. Plastic deformation pits in silicon nitride "A" that have been produced in 15 seconds of cavitation exposure. Optical microscope

Fig. 3. shows the measurement with an interferometer profiler of a plastic deformation pit produced in the first minute.

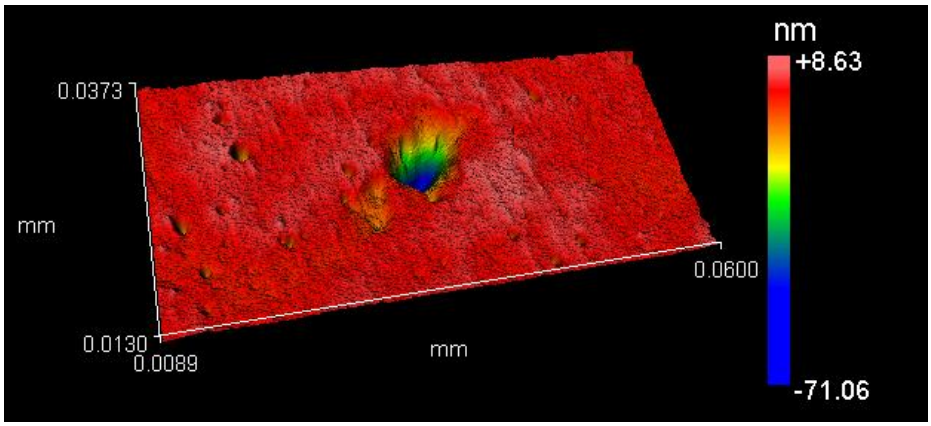

Fig. 3. Plastic deformation pit in silicon nitride “A” that has been produced in one minute of cavitation exposure. Interferometer measurement

Fig. 4. shows three plastic deformation pits seen with polarizing filter with two magnifications. They are indicated with circles. The contrast produced by the slope of the plastic deformation is very obvious at low magnification with polarizing filter but it is not so evident at high magnification, this is an optical effect. Conversely, it is possible to observe at high magnification that no material loss has occurred although there is some light crack on the surface due to plastic deformation compression.

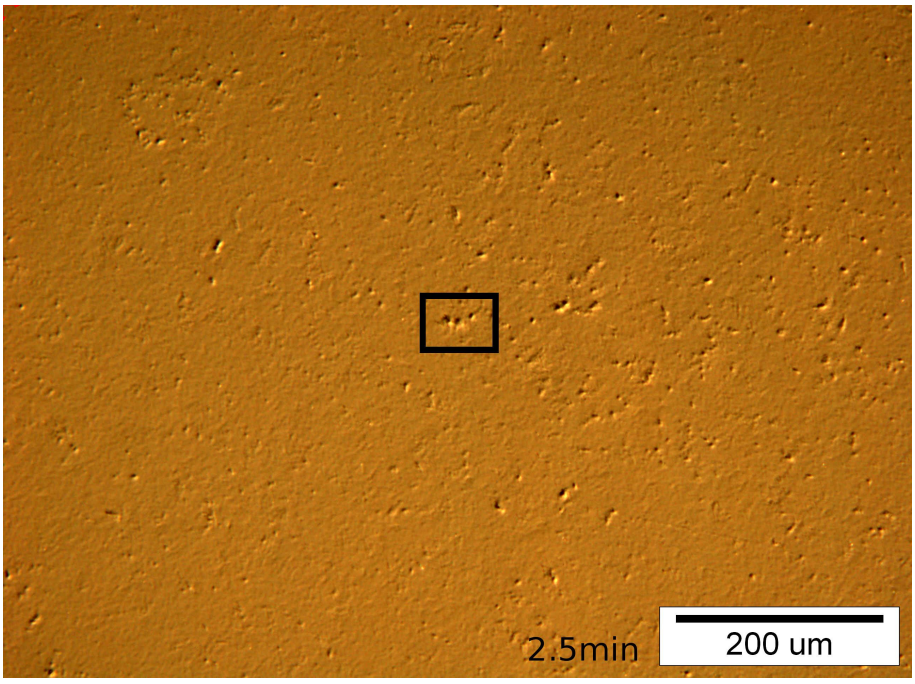

Fig. 4(a). Plastic deformation pits in silicon nitride “B” that have been produced in 2.5 minutes of cavitation exposure. Optical microscope

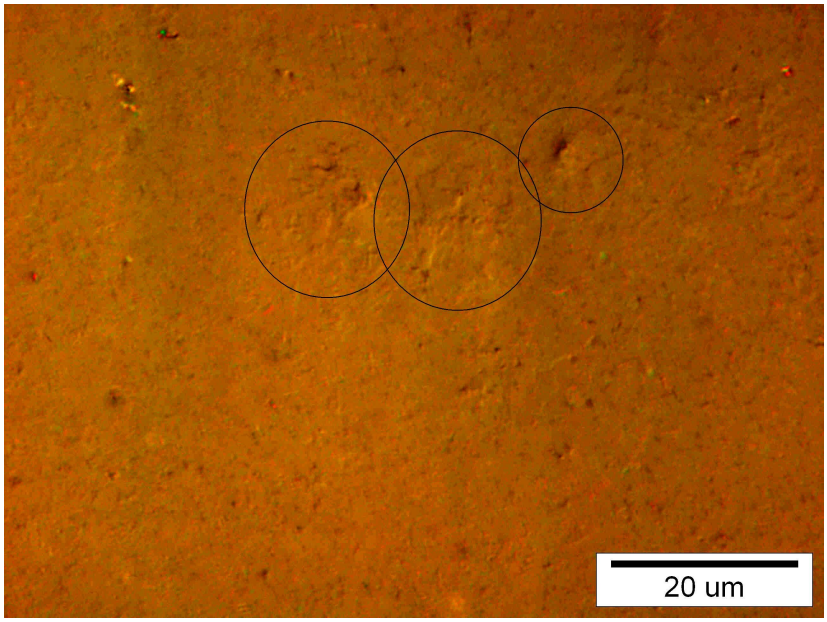

Fig. 4(b). High magnification of the indicated zone of Fig. 3(a). Optical microscope

Fig. 5. shows the surface of the silicon nitride “A” eroded after 180 minutes. Every spall out, river mark, crack and small pit on the surface has been produced by cavitation. Original surface was empty of these surface characteristics.

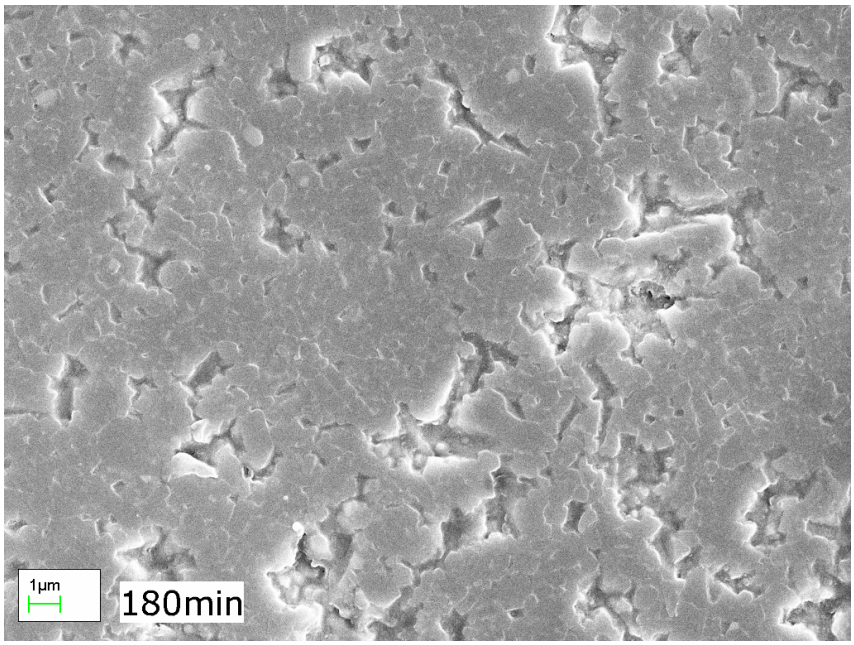

Fig. 5. Spalls out and river marks on a Thosiba silicon nitride after 180min.SEM.

Fig. 6 shows the spalls out of the silicon nitride seen with an optical microscope. It is possible to see the small spalls out but it is hard to see the cracks sown in the SEM photo. Fig. 5. The plastic deformation and the pressure impacts end to create the cracks and the spalls out shown in Fig. 5.

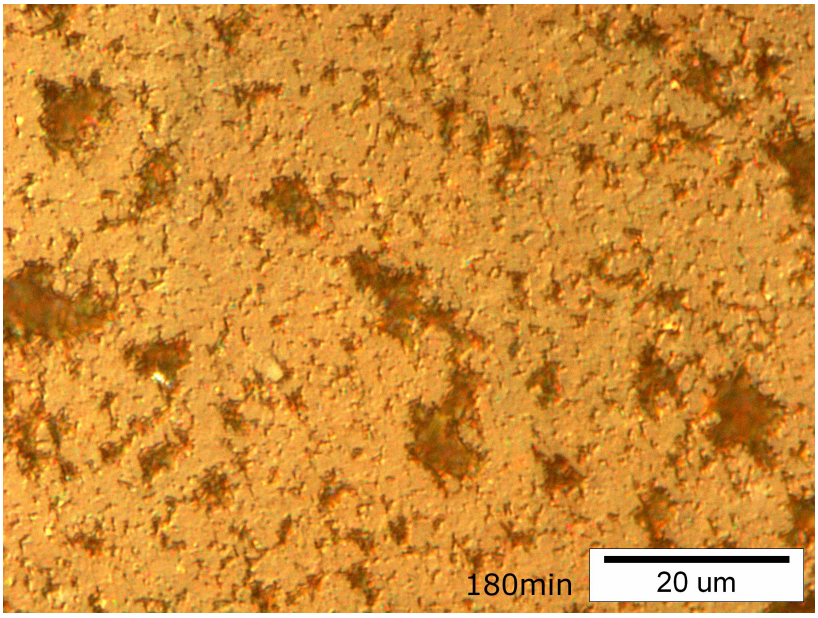

Fig. 6 Spalls out on silicon nitride “A” after 180min. Optical microscope

3.2 Zirconia.

Zirconia has the best cavitation erosion resistance behaviour among the materials studied. It is difficult to see deformation pits in the surface at the beggining, however, four small plastic pits have been seen in the first 30 seconds, and this number increase proportionally with the time. This plastic pits are similar to those shown on the surface of silicon nitride but they used to be smaller.

The process of material loss consists in the following: the pressure and the plastic deformation create cracks that with the time become river marks, because of smalls spalls out on the borders of the cracks. Then these cracks can produce a big spall out normally because of the joint of several in different sides of the new big spall out. Fig. 7. Fig. 8.

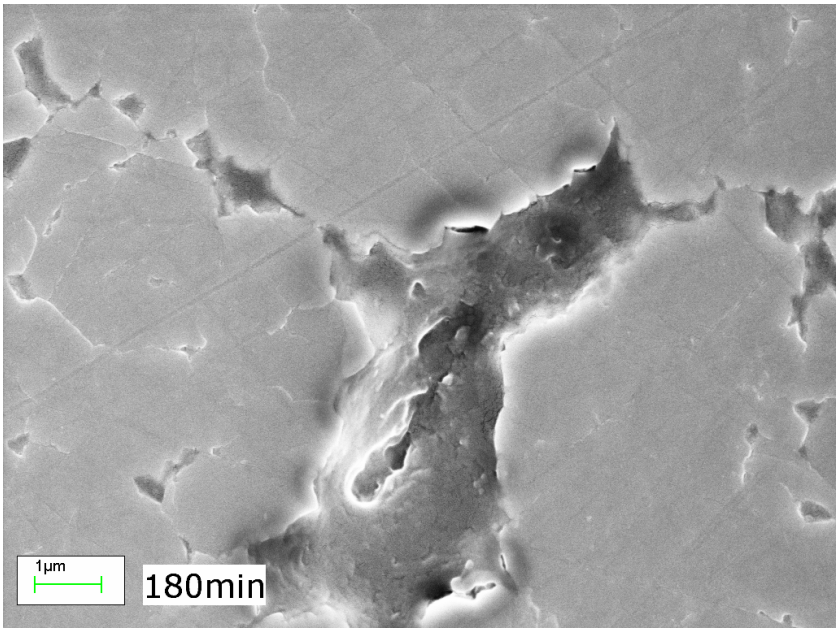

Fig. 7. Zirconia. Spalls out, cracks and river marks. SEM

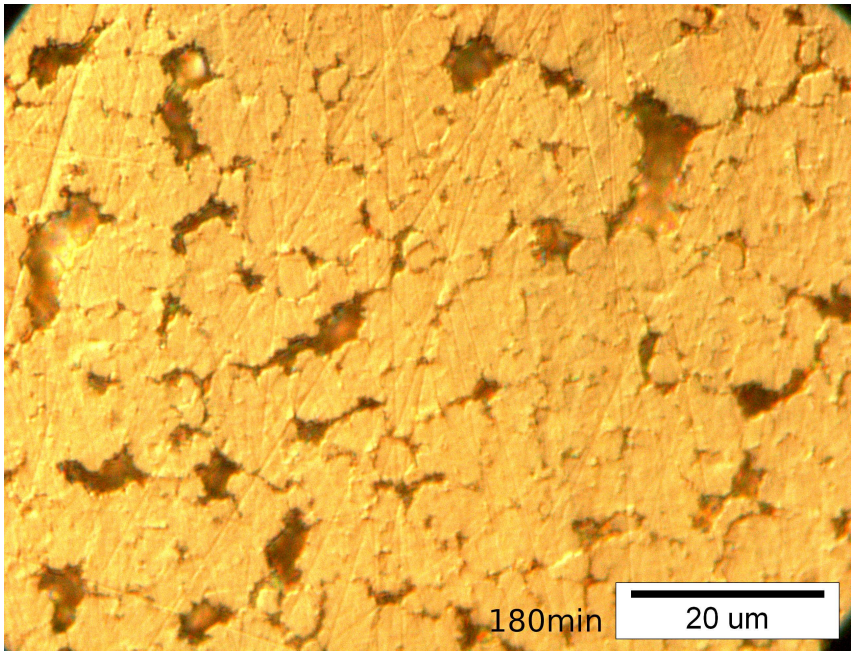

Fig. 8. Zirconia. Spalls out and river marks. Optical microscope.

Zirconia has a metastable phase that can change to other phase with the pressure. The tetragonal phase can become monoclinic with enough pressure. The relation of this with the cavitation resistance has been studied in [11].

The present study has found out that the surface can change just with the time. Fig. 9(a) shows the surface after 40 minutes of cavitation exposure. Fig. 9(b) shows exactly the same surface that Fig. 9(a) two months later. The roughness has increased, and the surface has now much more number of cracks that are visible with polarizing filter. The new surface has a topography similar to mountains. This shows that the metastable tetragonal phase need time to become monoclinic after being "activated" with cavitation. This transformation occurred at room temperature 20±8ºC.

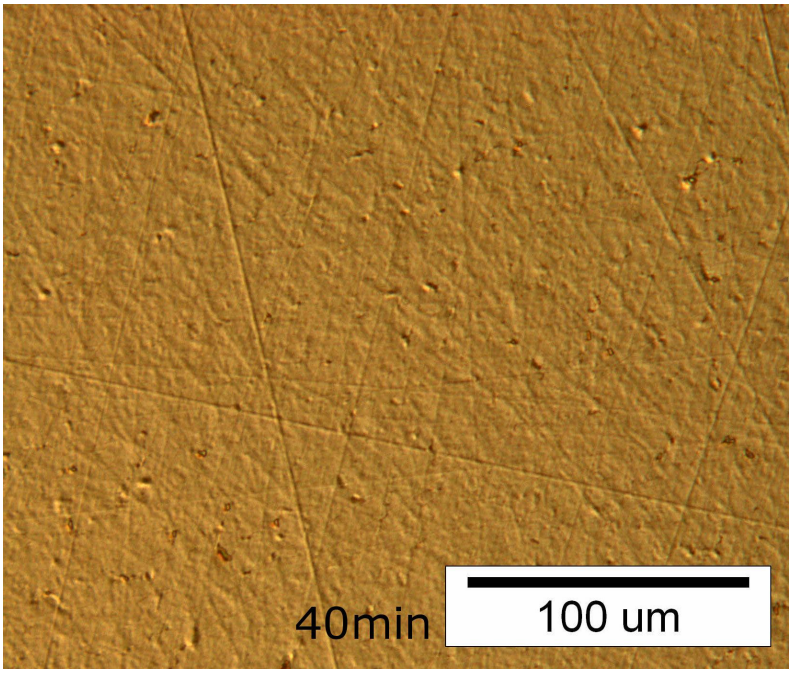

Fig. 9(a). Zirconia. Surface state after the experiment. Optical microscope.

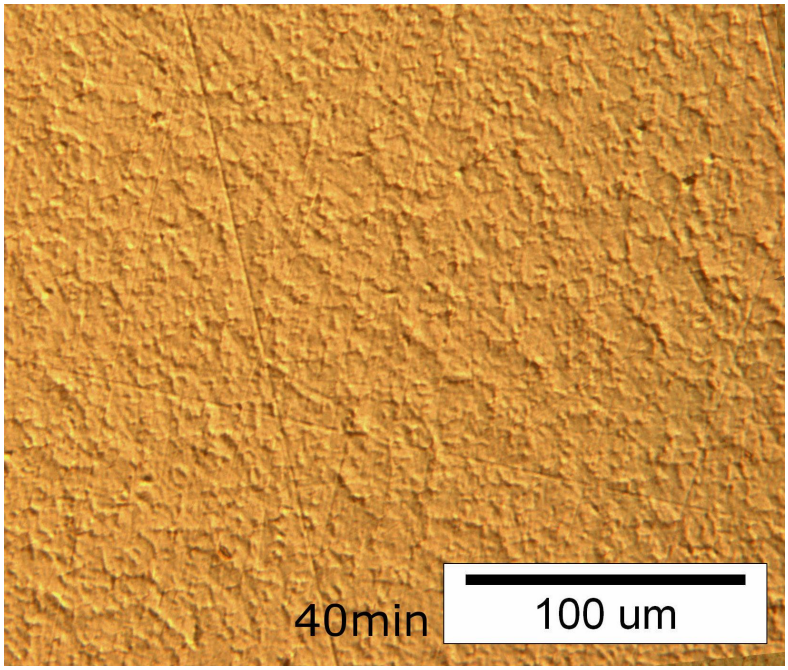

Fig. 9(b). Zirconia. Surface state 2 months after the experiment. Optical microscope.

The roughness of the sample has been measured in a region exposed to cavitation for 40 minutes one day after and eleven days after, and in other region exposed to cavitation for the same time two months ago. These values are shown in the table 2.

	R_a	*rms*
Region “A” after 1 day	0.004µm	0.006µm
Region “A” after 11 days	0.006µm	0.010µm
Region “B” after 2 months	0.008µm	0.011µm

Table 2. Zirconia, change of roughness with the time after 40 minutes of cavitation exposure. The surface has been “activated”.

Other regions of the zirconia sample have been exposed to cavitation for 120 minutes, these regions have the roughness shown in table 3. It looks like the transformation of phase restores the surface in some way, because the roughness is smaller after 11 days and after several months. These measurements have been taken with the white light interferometer. The resolution of the scanning was 0.22µm and the objective was 50x.

	R_a	*rms*
Region “C” after 1 day	0.032µm	0.158µm
Region “C” after 11 days	0.014µm	0.048µm
Region “D” after 2 months	0.019µm	0.065µm

Table 3. Zirconia, change of roughness with the time after 120 minutes of cavitation exposure.

3.3 Alumina.

Alumina cavitation erosion shows in the first 30 seconds a very few number of points with change of slope, this is not clear if it is due to plastic deformation or fracture and they are very small. Conversely, with the time, instead of plastic deformation, cracks grow, specially close to other cracks or holes from the finishing polish. These cracks develop in spalls out in the first minutes. Fig. 10(a). and Fig. 10(b). show the crack growth and the spalls out in the times 6 and 10 minutes. The mechanism of erosion of the alumina is completely different to the others materials.

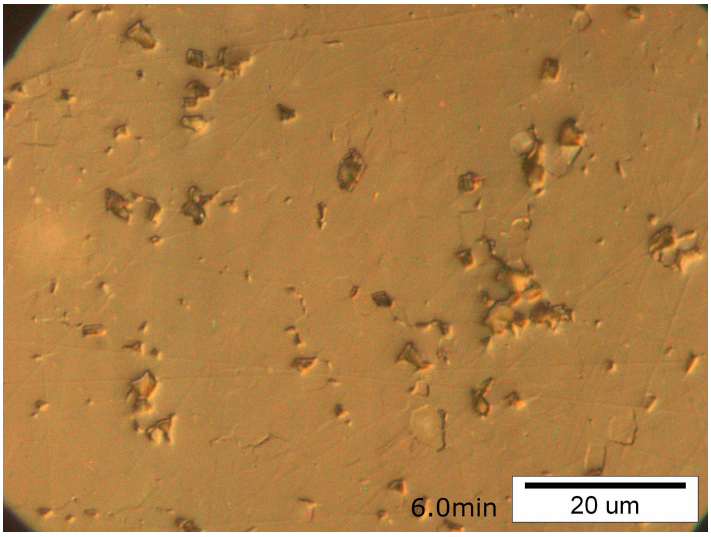

Fig. 10(a). Alumina. Cracks and spalls (6 minutes). Optical microscope.

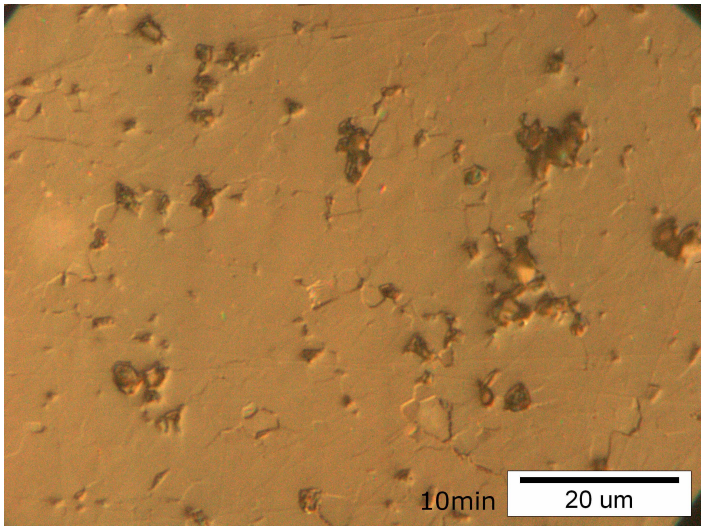

Fig. 10(b). Alumina. Cracks and spalls (10 minutes) . Optical microscope.

3.4 Stainless Steel.

Stainless steel has been exposed to cavitation erosion, the behaviour of this material is completely different to other due to its capability of deformation and work-hardened. The plastic deformation in its surface is much bigger, furthermore, it starts in the few first seconds to become the whole surface deformed. To see the plastic deformation pits isolated one from each other is necessary to stop the transducer in the first seconds. This study has done it after two seconds, in order to measure the volume of the pits respect the original surface.

The number of plastic deformation pits over a determined area increased quickly in the first minute, these pits were very big. After, the number of pits increased but these pits were smaller with the time. Fig. 11 shows this behaviour. This effect is produced because of the work-hardened of the surface produced by plastic deformation.

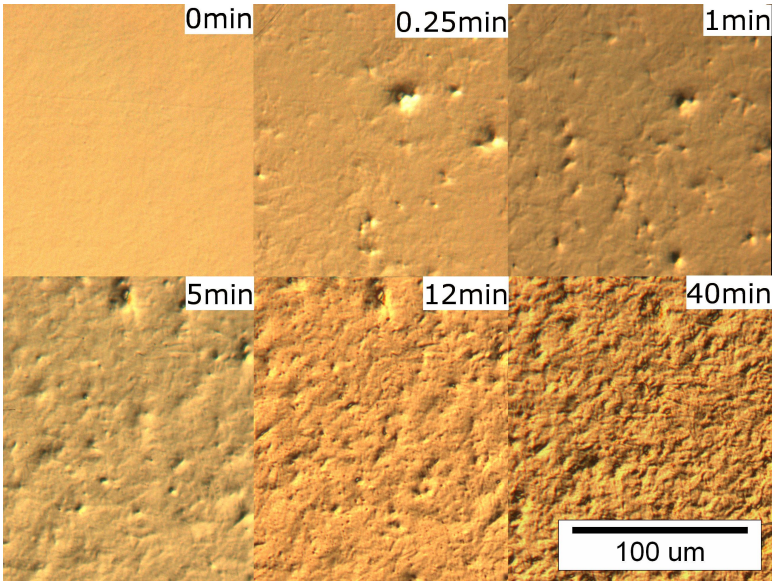

Fig. 11. Stainless steel. Plastic deformation. Optical microscope.

The typical form of the plastic deformation pit is shown in Fig. 12. The vertical magnification scale is much larger than the horizontal ones. This shows that there is no accumulation of material on the sides of the pit, thus, the pit is so shallow that the material, probably, has been just compressed towards inside of the surface, orthogonal to this.

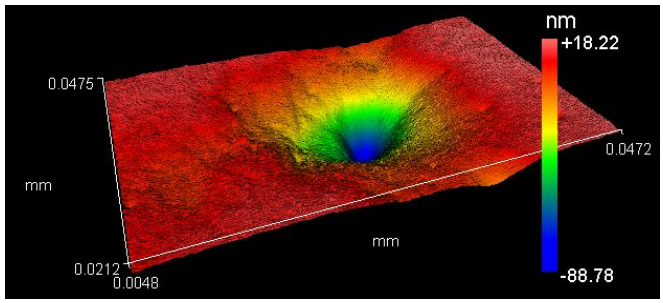

Fig. 12. Plastic deformation pit in Stainless Steel that has been produced in two seconds of cavitation exposure. Interferometer measurement.

A red colour has appeared in the region of more severe cavitation erosion during the second hour of cavitation exposure. Although it may be possible to think that this colour is due to oxidation because of the high temperatures that cavitation can achieve, the analysis of elements in the scanning electronic microscope has not showed evidence of oxygen in the region. The Fig. 13 shows this coloured effect.

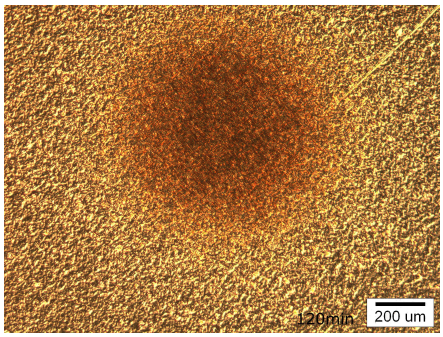

Fig. 13. Cavitation erosion region after 120 minutes of cavitation exposure. Optical microscope.

3.5 Rankings.

In order to create a ranking of the materials and to obtain a better understanding of the mechanisms of cavitation erosion of these materials, several criterions have been followed. Table 3. shows the volume and the depth of a typical big plastic deformation pit visible with polarizing filter and low magnification in optical microscope. It has been measured with interferometer profiler and maximum magnification and the result showed is an average of at least three different pits for each material. Some examples of these pits are Fig. 3 and Fig 12.

	Time of measurement	Typical Volume (μm^3)	Typical Depth (μm)
Alumina	-	Not measurable	Not measurable
Zirconia	4min	0.6	0.03
Silicon Nitride, “A”	1min	1.7	0.05
Silicon Nitride, “C”	0.5min	2.0	0.06
Silicon Nitride, “D”	1min	2.2	0.08
Silicon Nitride, “B”	2min	3.3	0.09
Stainless Steel	2sec	10.0	0.13

Table 3. Measurement averages of plastic deformation pits with profiler interferometer.

Table 4. shows the surface roughness after 120 minutes of cavitation exposure. The table has been ordered from smaller to greater roughness.

	R_a	*rms*
Silicon Nitride “A”	0.007µm	0.020µm
Zirconia (after 11 days)	0.014µm	0.048µm
Zirconia (after 3 months)	0.019µm	0.065µm
Zirconia (after 1 day)	0.032µm	0.158µm
Stainless Steel	0.153µm	0.206µm
Silicon Nitride “C”	0.527µm	0.670µm
Silicon Nitride “B”	0.654µm	0.913µm
Silicon Nitride “D”	1.083µm	1.324µm
Alumina	1.138µm	1.437µm

Table 4. Surface roughness after 120 minutes to cavitation exposure.

Table 5. shows the number of plastic deformation pits that have been possible to count using optical microscope with polarizing filter and low magnification after 30 seconds of cavitation exposure. In the case of alumina is not clear if this is plastic deformation or slope change due to fracture. This measurement has been made summing the pits produced in the first 15 seconds plus the pits produced from 15 to 30 seconds. The number of pits was similar in both times. The photos were aligned with an image software to recognise easily the new pits.

Material	Pits
Zirconia	4
Alumina	7
Silicon Nitride “A”	62
Silicon Nitride “B”	62
Silicon Nitride “D”	77
Silicon Nitride “C”	89
Stainless Steel	massive

Table 5. Number of plastic deformation pits produced in the surface after 30 seconds of cavitation exposure.

Table 6 shows the surface that has been removed due to cavitation erosion after 120 minutes of exposure. To calculate this, the photos have been processed with an image software. The photos that have been taken without polarizing filter have been transformed in photos of two colours, black and white, changing the brightness and the contrast, and the number of black pixels have been counted with the software. As the size of the pixel is known, the area of surface loss has been calculated. The processed photos are shown in Fig. 14. Table 7 shows the comparison of silicon nitride “A” and Zirconia in a later state, after 180 minutes of exposure. In this stage Zirconia has a better result.

Material	**Surface loss**
Silicon Nitride “A”	0.006 mm^2
Zirconia	0.015 mm^2
Silicon Nitride “B”	0.308 mm^2
Silicon Nitride “D”	0.450 mm^2
Silicon Nitride “C”	0.548 mm^2
Alumina	>1.067 mm^2

Table 6. Surface loss after 120 minutes to cavitation exposure.

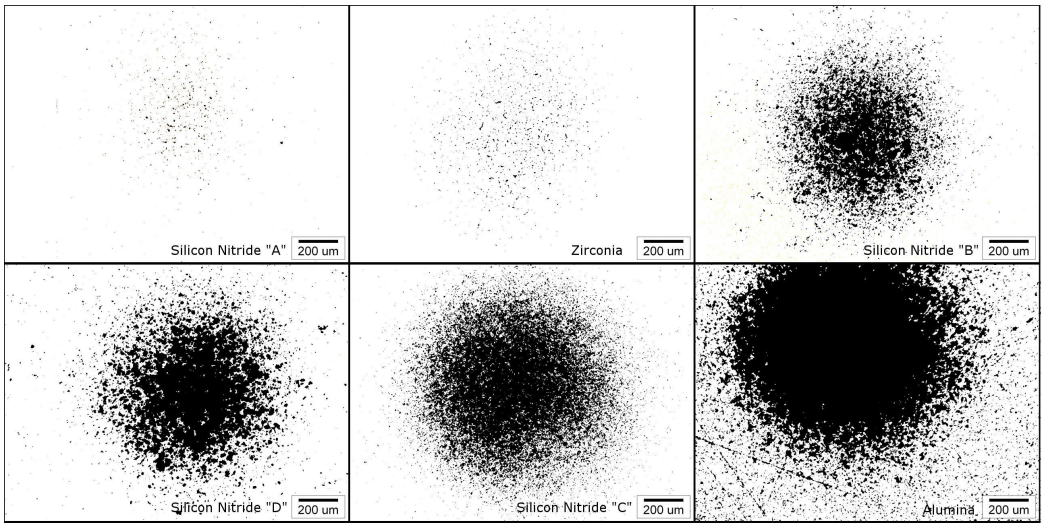

Fig. 14. Surface loss after 120 minutes to cavitation exposure.

Material	Surface loss
Zirconia	0.091 mm^2
Silicon Nitride "A"	0.144 mm^2

Table 7. Surface loss after 180 minutes to cavitation exposure.

4. Discussion

The initial roughness of the samples, table 1, shows that there are important differences in the behaviour of the material when it is eroded with a diamond slurry. The same method has been followed to polish the samples but the obtained results are different. Alumina, that is the most brittle, has greater roughness than the others. This important difference is a key factor in the cavitation resistance performance.

When ceramics materials are exposed to cavitation, plastic deformation pits are created in the surface. In the case of alumina, that is more brittle than the others, fracture occurs and material loss is produced, Fig 10. Although some small changes of slope has been seen in alumina, this is not clear if they are produced by plastic deformation, by fracture or by both. Silicon nitride and zirconia stand the plastic deformation but it looks like plastic deformation produce cracks around itself, Fig 4(b), these cracks grow with the time and then some small pits appears in the border of the cracks because of a small material loss. These small pits spread and become big holes like those shown in Fig 5, Fig 6, Fig 7 and Fig 8.

The phase change of zirconia, from the metastable tetragonal phase to monoclinic phase has a delayed. When the plastic deformation happens, a residual stress is produce inside the surface and this produce the change of phase with the time. At room temperature it takes several weeks. This effect produced confusion with the results related with zirconia, because two different effects play an important role: the cavitation erosion itself and the change of volume produced by the toughening transformation. It has been demonstrated that the toughening transformation produces a change of the surface characteristics with the time, without more cavitation exposure. This effect has increased the roughness at 40 minutes of cavitation exposure but it has decreased the roughness at 120 minutes of cavitation exposure (table 2 and table 3). For this reason it is necessary to fix what kind of behaviour we are looking for in the zirconia. If the application is a short time application, like several minutes or hours, it is necessary to test the material without delays. But if the application is long term, like months or years, it is necessary to test the material with these delays, trying to simulate the length of the normal life of the component.

In the case of stainless steel, it looks like that big bubbles collapsing close to the surface created the big pits at the beginning, when the surface was not work-hardened. While this happens, other bubbles not so big or not so close collapsed as well and produced plastic deformation on the surface, but this was not in form of pit, but in a more extended form all over the surface. The result was a work-hardened surface with pits whose size depends on the flux of energy of the collapsing bubble and the local state of the surface. Fig 11. The important contribution of this to the understanding of the behaviour of the other materials is that there is a big range of severity in the energy flux of the bubbles collapsing close to the surface. In table 3 the volume that is measured is the volume of the typical big pits, those that are easy to see with optical microscope and interferometer profiler. Table 5 shows that the number of damage produced by cavitation is much smaller in zirconia and alumina. This means that only the collapsing of bubbles that hit with an amount of energy above a threshold creates a permanent effect on the surface. This threshold is higher for zirconia and alumina than for silicon nitride.

Alumina has a small number of damages in the first 30 seconds, conversely it is the material with the poorest cavitation resistance. Silicon nitride "A" has a big number of damages in the first 30 seconds, similar to the others silicon nitrides, but it has much better

cavitation resistance in terms of keeping the surface, table 6. These facts evidence that the cavitation resistance is related with: a) how the materials stand the flux of energy of the collapsing bubble and b) how the material avoids the propagation of the cracks and the spread of the small pits in the border of the cracks. The mechanical characteristics of the material may play a secondary role in comparison with the microscopic mechanical characteristics of grains and intergranular material.

Zirconia has demonstrated the best cavitation resistance, followed by one of the commercial silicon nitrides, this silicon nitride is believed to have different microstructure than the others. Alumina has the poorest cavitation resistance.

6. Conclusions

Although it is widely believed that the cavitation exposure of ceramics can not produce plastic deformation or this is negligible, plastic deformation pits have been detected with polarizing filter with optical microscope and have been measured with interferometer profiler in silicon nitride and in zirconia. In alumina this has not been possible.

Plastic deformation pits play a key role in the way of the material to resist cavitation erosion. Alumina, that can not absorb very well energy through plastic deformation, suffers fracture from the first moment that soon become spalls out. Conversely the absorption of energy by means of plastic deformation in silicon nitride and zirconia allows delaying the creation of big fractures and cracks that cause the material loss.

The change of phase in zirconia from metastable tetragonal to monoclinic with the correspondent volume increase is not an instantaneous effect, but this effect takes several weeks to happen after being exposed the surface to cavitation at room temperature. In the zirconia that has been studied, big changes have been seen after two months. This effect, known as transformation toughening, is caused by the pressure. When the surface is exposed to cavitation, plastic deformation is produced on the surface, this induces residual stress. This residual stress “activates” the surface, that leads to the change of phase in the future.

Plastic deformation pit size in stainless steal depends on the local state of the surface due to the work-hardening produced by plastic deformation caused by bubbles that have collapsed before.

Intergranular material and the way that it can stand plastic deformation in the surroundings and in itself have a fundamental role in the cavitation erosion resistance performance. How the intergranular material and the joint of this to the grain respond to the plastic deformation is thought to be the principal cause of the different performances of silicon nitrides from different manufacturers. (This has not been demonstrated in this study).

References

[9] ASTM G32-03, Standard Test Method for Cavitation Erosion Using Vibratory Apparatus, ASTM, 2003

[1] W.J. Tomlinson, S.J. Matthews, Cavitation erosion of structural ceramics, Ceramics International 20 (1994) 201-209

[2] W.J. Tomlinson, N. Kalitsounakis, G. Vekinis, Cavitation erosion of aluminas, Ceramics International 25 (1999) 331-338

[3] D. Niebuhr, Cavitation erosion behavior of ceramics in aqueous solutions, Wear 263 (2007) 295-300

[7] A. Moussatov, C. Granger, B. Dubus, Cone-like bubble formation in ultrasonic cavitation field, Ultrasonics Sonochemistry 10 (2003) 191-195

[8] A. Moussatov, C. Granger, B. Dubus, Ultrasonic cavitation in thin liquid layers. Ultrasonics Sonochemistry 12 (2005) 415-422

[4] C. Haosheng, L. Jiang, C. Darong, W. Jiadao, Damages on steel surface at the incubation stage of the vibration cavitation erosion in water, Wear 265 (2008) 692-698
[5] C. Haosheng, L. Shihan, Inelastic damages by stress wave on steel surface at the incubation stage of vibration cavitation erosion, Wear (2008), doi:10.1016/j.wear.2008.05.011
[10] C. Haosheng, L. Jiang, L. Fengbin, C. Darong, W Jiadao, Experimental study of cavitation damage on hydrogen-terminated and oxygen-terminated diamond film surfaces, Wear 264 (2008) 146-151
[11] J. Lu, et al., Microstructural effects on the resistance to cavitation erosion of ZrO_2 ceramics in water, Wear (2008), doi:10.1016/j.wear.2008.04.028
[6] F. Ronald Young, Cavitation, Imperial College Press 1999, ISBN 1-86094-198-2