Behavior of Polymer Thin Films Deposed on Bronze Surfaces at Cavitation Erosion

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One of the most actual problems regarding reparation and reconditioning of ships propellers surfaces or of some parts of hydraulic machines (turbines, pumps) is that of the erosion produced by cavitation, both from technical difficulties and costs. That is why researchers and builders are searching for new cheap solutions mainly regarding these surfaces protection. The present study concerns the analysis of polymer thin films meant to protect surfaces of bronze parts exposed to cavitation. The bronze is a material frequently used to cast maritime or fluvial vessels propellers. The test, conducted at Timisoara Polytechnic University Cavitation Laboratory on standard vibrating equipment, showed the poor resistance of polymer films and revealed the necessity of continuation the studies especially in finding new techniques in order to increase the polymer films adhesion to the metallic surfaces that had to be protected.

Keywords: polymers, collagen, cavitation erosion, pitting, SEM analysis

During the last years the polymers and their composites applications are diversifying due their properties that can be designed. The composites might be regarded as structures with special properties due the fact that they are formed from different materials [1-3]. From these category, composite materials with thermo set matrix (especially epoxy resins) due to the modifications of their properties that can be obtained by using soft-ware applications [4, 5] or advanced processing technologies [6] are subject of intense researches in order to be used as protecting films for details working in abrasive erosion or cavitation erosion condition. In the present there are not known details of the composites fulfilling the above mentioned conditions, but there is known for instance that polymer materials as Belzona 1341 [3] and Belzona 2141 [7] are used to repair or protect surfaces subjected to cavitation erosion (Banki turbine runner, inside channel of spiral chamber, inner channels of water transportation pipes). These already existing applications and the results obtained along 60 years of researches [8,-11] are reasons to amplify the researches in order to find the best solutions of composites, polymers or modified polymers, able to resist to high intensity cavitation load as the ones produced by ship propellers or those produced by runners of hydraulic turbines or impellers of pumps. This are the reasons determining the researches to study the use of polymers or their composites as covering protecting films or as repairing agents for abrasive and cavitation erosion affected areas aiming to find cheap solutions and diminishing repair costs.

Experimental part

Polymers

Based on the idea of developing local chemical reactions inside a liquid phase polymer or pre-polymer (as in the case of citric gels method) [2-6, 12, 13], a mixture of epoxy resin and a modified epoxy-novolak vinyl-ester resin had been modified by using various organic and inorganic agents. The final obtained mixture of polymers was used to form thin films on bronze surfaces aiming to protect the metal against the erosion and especially the cavitation one.

Even if they are used mostly as industrial adhesives, the epoxy resins are generally brittle materials, easily failing when subjected of random loads, as the cavitation micro-jets or shockwaves, losing their integrity and as a consequence, their protective capacity. From this point of view the polymer used in this study is a mixture of an epoxy resin and a vinyl-ester one who might show a better flexibility relative to epoxy resin and being more suitable for metal protection. The mixture was used to cover, in the form of a thin film, only the metallic surfaces subjected to cavitation effects considering that the epoxy resin will ensure the adhesion to the metallic surface and the vinyl-ester resin will ensure the film flexibility. The polymer mixture was also modified by using a collagen–organic agent meant to increase de flexibility due its ability to form its own flexible structures and some inorganic agents ($BaCl_2$, $CuCl_2$, YCl_3 and $ScCl_3$) meant to develop nanostructures of mixed metallic oxides to improve the composites properties.

Mean time the presence of inorganic compounds, with nano dimensions, leads to local modifications of polymer properties (crystallinity, hardness) and they could lead to a better adhesion of polymer to the metallic surface (metal-metal interaction) with consequences regarding a better metal surface protection by increasing the unsoldering resistance respectively to the local fatigue loads induced by cavitation the micro-jets.

For this study we used epoxy resin *Epiphen RE4020* [14] with the hardener *DE4020* (*Resoltech*) and Vinyl-ester, epoxy novolac resin, diluted in styrene, with high reactivity and medium viscosity - *SIRESTER VE 64-M-140* (*Sir Industriale SPA*) [15]. The epoxy resin has a gel time of 45 min (in large volumes) and seven days for complete polymerization (in order to facilitate the presentation the epoxy resin will be denoted as Epiphen). The second resin (denoted as Sirester) has a gel time of about 20 min. The mentioned gel times are for large volumes of polymer and they are irrelevant for thin films or to controlled conditions of polymerization (for instance a higher value of temperature relative to the recommended value of this parameter).

From previous researches it was known that the inorganic substances solubility into the two components of Epiphen (RE4020 and DE4020) was very poor. Some tests showed that the solubility of these inorganic

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compounds into Sirester is higher, so the mixture of the two polymers could lead to higher solubility levels. Therefore, these facts encouraged the research development in two directions: one regarding the protective thin films for metallic surfaces and the other regarded mixed metallic oxides nanostructures (inside the polymer mixture). The presence of both organic and inorganic agents inside the polymer mixture may have effects on the gel time and on the polymerization time but these two aspects were not taken into account in this study.

The polymer mixture (modified with organic and inorganic compounds) was used to cover bronze (10.46 % Al, 4.85% Ni, 4.72 %Fe, 1.41 % Mn, the rest being Cu) parts what are denoted with A (two samples A1 and A2) and B (two samples B1 and B2) and these notations were kept also for the cavitation tested specimens.

Polymer preparation

The procedure followed to obtain the polymer mixture is listed below and it is a long term process.

1. Into two pots 100g Siretster and 5g collagen and the mixture were stirred for an hour (on a magnetic stirrer) at a speed of 500 rot/min and a temperature of 50°C;
2. In each of the two pots amounts of 4.165g of $BaCl_2$ and stirring was continued for another hour;
3. Amounts of 4.03g of CuCl2 were added into the two pots and the stirring was continued for one week (with the same stings of the magnetic stirrers - 50°C and 500 rot/min);
4. Because of the presence of precipitate into the pots 100g of Sirester were added in each pot and the stirring was continued for another week;
5. Into the first pot (A) 1.953g of $ScCl_3$ were added while into the second pot (B) 1.953g of YCl_3 were added and the stirring was continued for another week;
6. A quantity of 200g Epiphen (RE4020) was added in each pot and stirring was continued (with the same settings) for six weeks obtaining homogeneous mixtures in both pots.

Thin films application on the metallic surfaces

Prior to effective film application each metallic piece had been oxidized on its plane and cylindrical surfaces (fig. 1).

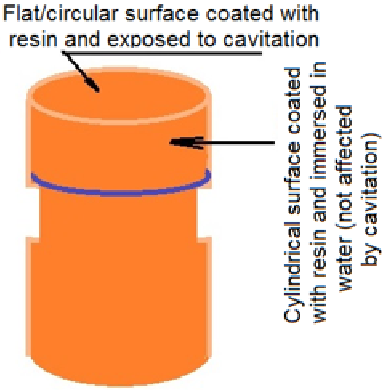


Fig. 1 Image of the cavitation specimen with showing exposed cavitation area and those coated with epoxy resin

The procedure consisted of wetting each piece with ethanol and followed by covering with citric acid (citric acid crystals adhered to the metallic surface due the presence of ethanol liquid film). The samples were then placed into an oven at 100°C where the metallic surfaces were oxidized as a consequence of citric acid combustion. After their natural cooling the metallic surfaces were polished with abrasive paper (the final roughness being Ra=20-25μm) then all the samples were washed with distilled water and 99% ethanol.

The application of the thin films consisted, in fact, of application of three successive layers of polymer and it had been realized with a laboratory rod mixer (generally used to mix various substances). Each metallic sample was fixed on a special rod and kept into the oven at a temperature of 90°C.

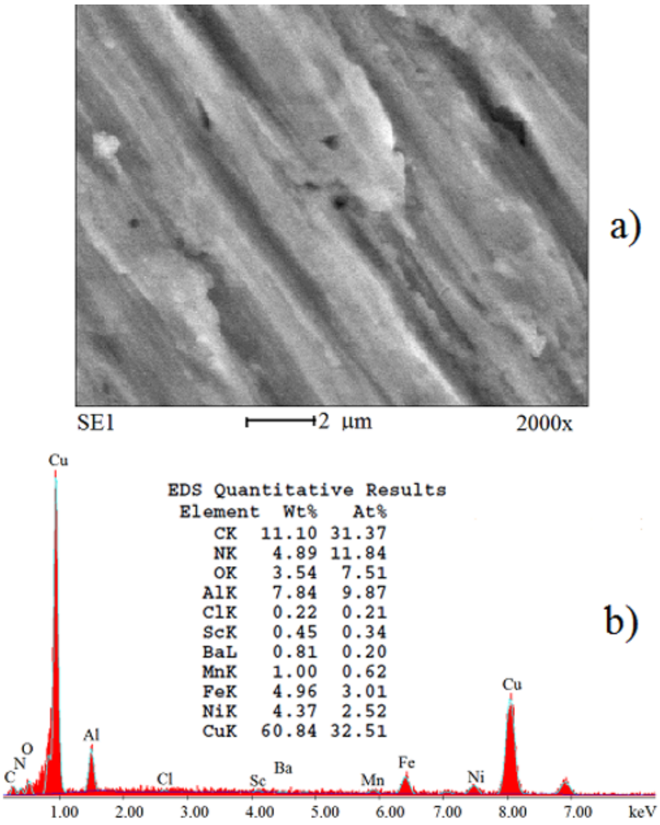
1. The special rod (with the attached specimen) is fixed in the mixer mandrel such as the specimen to be placed into the pre-polymer (its plane and cylindrical parts); the mixer is set for 50 rot/min and the specimen is rotate for 5min into the pre-polymer mixture.
2. The specimen is taken out from the polymer mixture and with the mixer set for 1000 rot/min is rotated in order to remove the polymer excess by centrifugation.
3. The cylindrical part (together, of course, with the plane one) is introduced into the Epiphen hardener (DE4020) and is rotated with 50 rot/min for 3 min.
4. The excess of hardener is removed by centrifugation at 1000 rot/min (out of the hardener volume).
5. The port specimen rod (with the attached specimen) is introduced into the oven at 90°C and kept for 45 minutes and then all the steps from 1 to 5 are repeated.

Because of the elevated value of the temperature of the sample it is expected as a part of Sirester to polymerize immediately after the specimen immersion into the polymer liquid mixture so it is fixed on the metallic surface and is engaging constituents of Epiphen (RE4020). The following immersion into the Epiphen hardener (DE4020) is ensuring (due the elevated value of temperature and due to the much reduced thickness of the pre-polymer film) the partial polymerization of Epiphen while the elevated value of temperature into the oven ensures an accelerated polymerization. There is a difference between the two samples denoted as 1 and 2 and this difference consist of polymerization of the second layer of polymer. In the case of the two samples denoted as A2 or B2 this middle layer had polymerized at the laboratory temperature for 45 min and, in this case it is expected that this two samples to show a better resistance at variable loadings due to the increased flexibility of this layer. Indeed, the behavior of the two types of samples (1 or 2) for each type of polymer mixture (A or B) is different as it will be seen later.

Morphological analysis

The basic goal of performing morphological analysis was to identify the existence of some nano dimension structures inside the polymer film and by means of SEM, some aggregates were observed and measured. As it easily can be seen in figures 2a, 3a, 4a and 5a there are not notable differences between the SEM aspect of analyzed samples, nor between the same polymer mixture (1 or 2 type probes) nor for the two types of polymer mixtures (A or B type probes). Together with SEM analysis the EDAX analysis is meant to identify the elemental distribution and, if necessary, the quantitative analysis of materials. SEM analysis reveals the existence of some nano dimension aggregates while the EDAX analysis is strongly influenced by the presence of massive metal in the bronze sample.

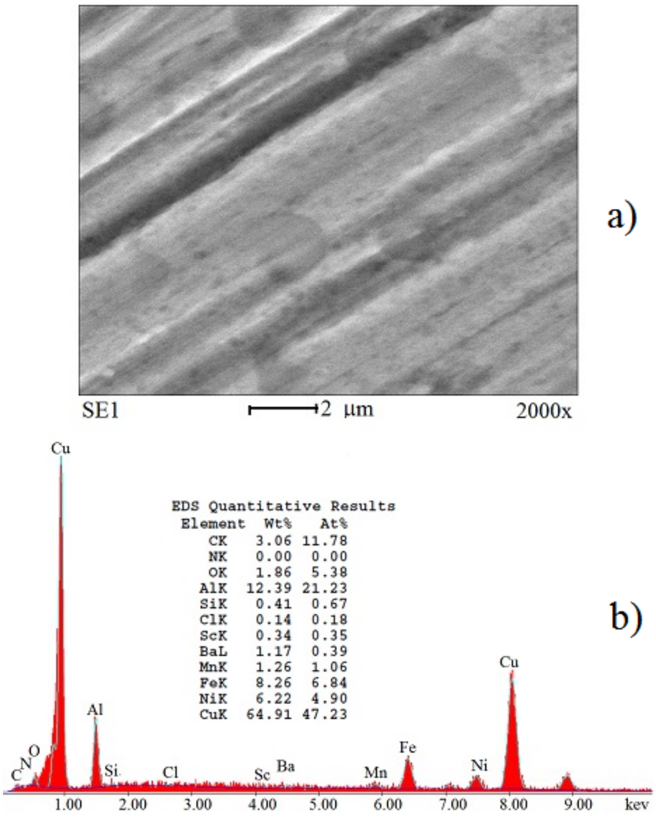
Whatever the qualitative analysis shows the massive presence of Copper (the main component of the metallic alloy) and the presence of relatively high concentrations of Iron, Manganese, Nickel and Aluminum, all of them being listed in the alloy receipt. As expected the presence of Barium, Scandium and Yttrium is low but that is explainable due their low concentration with respect for the metals that are parts of the alloy as it can be seen in figures 2b, 3b, 4b, and 5b.



EDS Quantitative Results

Element	Wt%	At%
CK	11.10	31.37
NK	4.89	11.84
OK	3.54	7.51
AlK	7.84	9.87
ClK	0.22	0.21
ScK	0.45	0.34
BaL	0.81	0.20
MnK	1.00	0.62
FeK	4.96	3.01
NiK	4.37	2.52
CuK	60.84	32.51

Fig. 2. Images SEM (a) and EDAX analysis (b) of epoxy resin A1, thin films

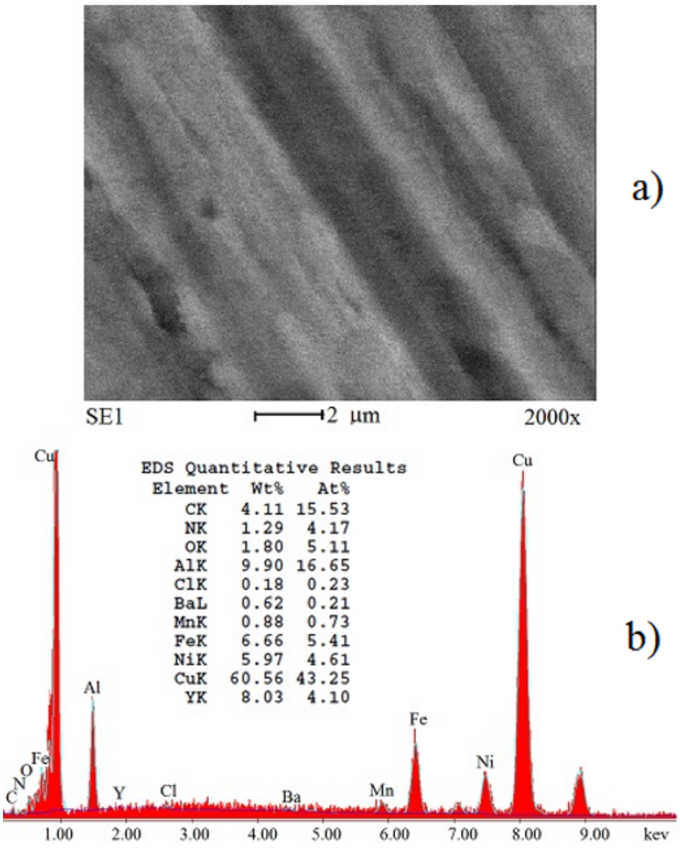


EDS Quantitative Results

Element	Wt%	At%
CK	3.06	11.78
NK	0.00	0.00
OK	1.86	5.38
AlK	12.39	21.23
SiK	0.41	0.67
ClK	0.14	0.18
ScK	0.34	0.35
BaL	1.17	0.39
MnK	1.26	1.06
FeK	8.26	6.84
NiK	6.22	4.90
CuK	64.91	47.23

Polimer 1.2

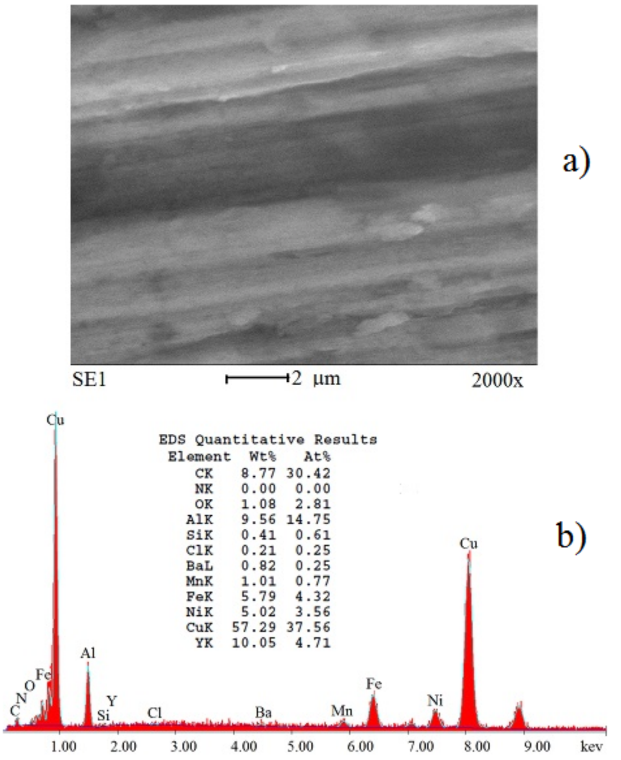
Fig. 3. Images SEM (a) and EDAX analysis (b) of epoxy resin A2 thin films



EDS Quantitative Results

Element	Wt%	At%
CK	4.11	15.53
NK	1.29	4.17
OK	1.80	5.11
AlK	9.90	16.65
ClK	0.18	0.23
BaL	0.62	0.21
MnK	0.88	0.73
FeK	6.66	5.41
NiK	5.97	4.61
CuK	60.56	43.25
YK	8.03	4.10

Fig. 4. Images SEM (a) and EDAX analysis (b) of epoxy resin B1, thin films



EDS Quantitative Results

Element	Wt%	At%
CK	8.77	30.42
NK	0.00	0.00
OK	1.08	2.81
AlK	9.56	14.75
SiK	0.41	0.61
ClK	0.21	0.25
BaL	0.82	0.25
MnK	1.01	0.77
FeK	5.79	4.32
NiK	5.02	3.56
CuK	57.29	37.56
YK	10.05	4.71

Fig. 5. Images SEM (a) and EDAX analysis (b) of epoxy resin B2, thin films

Also it easily to notice that from a EDAX analysis to another there are some variations between the concentrations of the same element but that is due to the fact that the scanned area is very small (in this case of about 80 µm^2) and at this level some aspect differences between different areas of bronze are observable. At this level the inhomogeneous distribution of metals into the alloy are significant. In all these analysis it is impossible to separate Copper from the alloy by the Copper placed into the polymer and, of course the height of the Copper peaks is determined by the Copper from alloy. The quantitative analysis reveals the presence of organic elements such as

Carbon, Oxygen, Nitrogen, Chloride all of them being present into the polymer mixture but, regarding the Oxygen; it can be also present in the form of mixed metallic oxides (resulted from the local developed chemical reactions).

The Raman analysis does not show chemical modifications, as it can be observed in figures 6b, 6c, 7b and 7c even their absence is not certain due their extremely low concentration which is make them undetectable and unobservable. On another hand the Raman microscope images are taken from a fracture area of a polymer bulk. Some baguettes of modified polymer had been formed in order to mechanically characterize them.

In figure 6 and figure 7 it can be seen that the two spectra corresponding to the bulk polymer mixtures show a peak at 1500 cm^{-1} and another one at 3000 cm^{-1}. In the case of thin polymer films the two spectra show the same profile but with a multiple peak centered at 1500 cm^{-1}. In this case it could be about the Raman print of the metallic substrate (unfortunately it was impossible to take out the Raman print of metallic alloy because at that time an uncovered sample of bronze was not available).

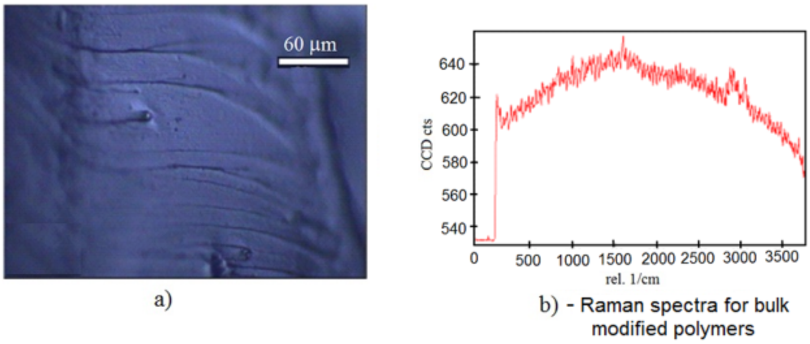
As well as the EDAX analysis, the results of Raman analysis are strongly dependent on the selected area and, as a consequence, they are of statistic nature and it is practically impossible to select the same area both for EDAX and Raman analysis. With these is clear why it is very difficult to point out the nanostructures but still the probable presence of mixed metallic oxides can produce a better bond between metal surface and the polymer film – in this respect the research has to be continued.

Cavitation tests

Equipment and methodology

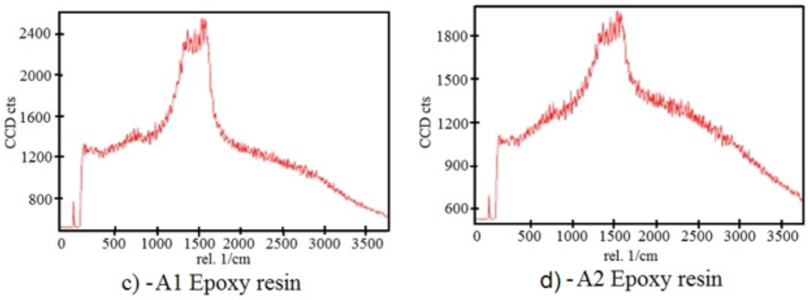
In order to prove the resistance of the thin polymer film to cavitation the standard vibration apparatus with piezoelectric crystals was used at the Cavitation Laboratory of Timisoara Polytechnic University [16,17]. The functional parameters of the apparatus were kept at usual values and the specific procedure of tests is the one listed in ASTM G32-2010 [18] and became a custom of the laboratory [16, 17, 19, 20]. The procedure includes sample preparation before and after tests, determination of mass lost, taking pictures of affected surfaces.

The test conditions imply test of 165 min long [17, 21]. In this case, due to the presence of the thin film with an unknown behavior, the tests duration were established by



a)

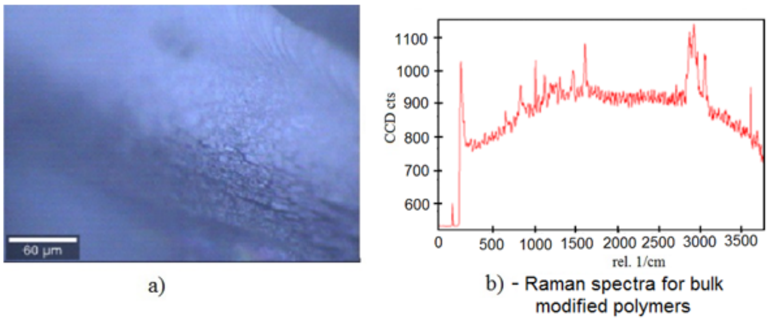
b) - Raman spectra for bulk modified polymers



c) -A1 Epoxy resin

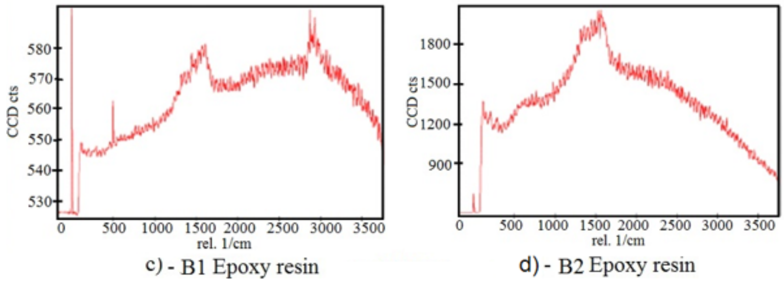
d) -A2 Epoxy resin

Fig. 6. Raman microscope images (a) and Raman spectra (b, c and d)



a)

b) - Raman spectra for bulk modified polymers



c) - B1 Epoxy resin

d) - B2 Epoxy resin

Fig. 7. Raman microscope images (a) and Raman spectra (b, c and d)

the appearance of pitting on the metal surface and the detachment of the entire polymer film.

Results and discussions

The cavitation micro-jets strongly affected the polymer thin film and it resists no more than 45 min perhaps due to the low adhesion to the metallic surface. So the standard evaluation based of specific curves and the comparison between characteristic parameters (mean depth erosion and mean depth erosion rate) was not possible. In this special case the evaluation of vibratory cavitation resistance of the polymer thin film is performed on the bases of the images taken with high resolution cameras and optical microscopes.

Images in figure 8 show the behavior of A1:

- after 5 min of exposure, at the specimen periphery some small pits appears and unsoldering of the thin film (on small areas) with water penetration between the metallic surface and polymer film;

- after 14 min of exposure the sample had broken and the test continuation was impossible but it may observed that the unsoldered area is increased (without the breaking of the thin film) and on the metal surface some small pits appears.

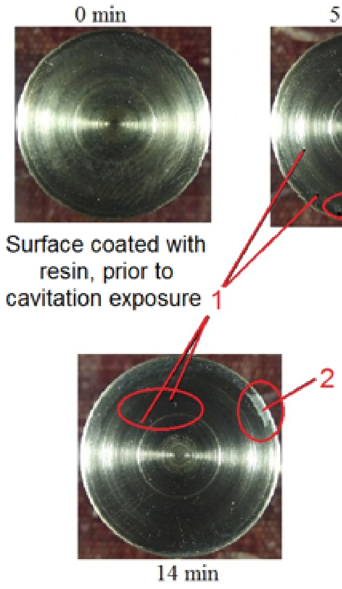


Fig.8. Behavior evolution to vibratory cavitation of the epoxy resin layers A1 deposited on the flat surface of the sample
1. perforations of the resin layer (cracks/pits);
2. detached resin film and bubbles with small dimensions

Images in figure 9 show the behavior of the A2 sample (the one we considered more flexible due to the natural polymerization of the second layer):

- after 5 min of cavitation exposure, the affected surface shows small pits and low traces of unsoldering without total disappearing of the film;

- after 15 min of exposures some new exfoliation areas appear toward the central zone, some cavities are observed but the depth of these cavities does not increase as in the case of metallic surfaces exposure;

- after 30 min the film presents large unsoldered areas especially at the periphery of the tested area and in smaller measure also in the central zone;

- after 45 min of cavitation the polymer film was removed from the metallic surface and caverns started to form in the zone of peripheral ring as is presented in [20].

Images in figure 10 present the behavior of B1 sample (again a rigid thin film):

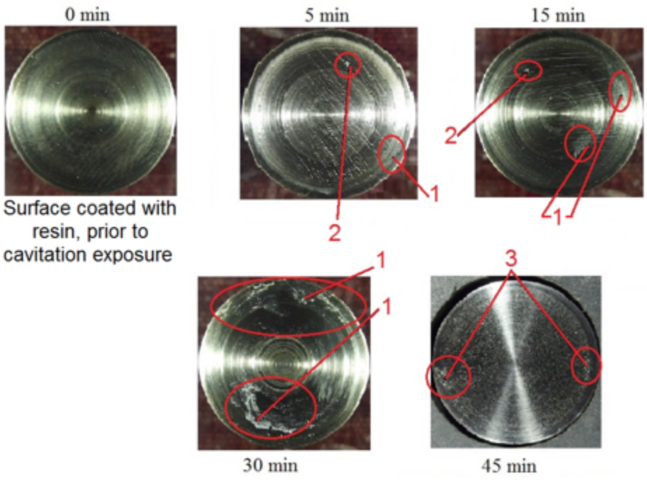


Fig.9. Behavior evolution to vibratory cavitation of the epoxy resin layers A2 deposited on the flat surface of the sample
1.detached resin film and bubbles with small dimensions;
2. cracks/pits; 3. caverns

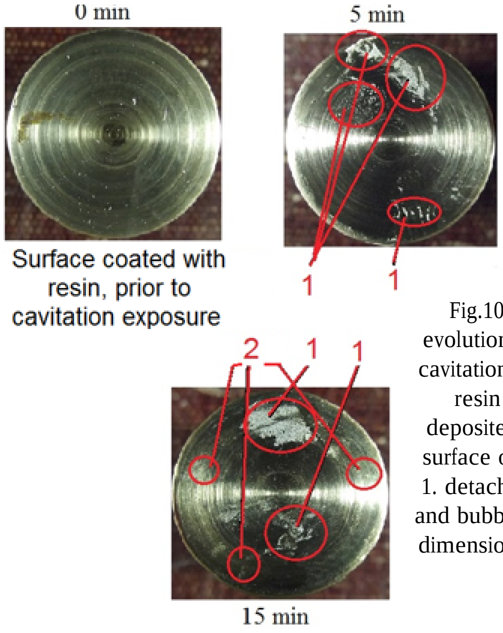


Fig.10. Behavior evolution to vibratory cavitation of the epoxy resin layers B1 deposited on the flat surface of the sample
1. detached resin film and bubbles with small dimensions; 2. cracks/ pits

- after 5 min of cavitation erosion some small areas of film unsoldering are observable both in the central zone and at the periphery. A large piece of film is detached from the edge to the center. Some pinching is also observable showing that in those places the film was ruptured. The aspect of these detachments offers the sensation of some voids similar to the ones appearing in the case of metal casted samples. The dispersion in the plane of the eroded area, in spiraled forms, is the effect of cavitation cloud similar to the one generated by the ships propellers or hydraulic machine rotors which is described in [16];

- after 15 min of cavitation attack the unsoldering surface is increased, in the surface plane new unsoldered zones are present. As in the case of A1 sample, in this case of B1 sample during the first seconds of the third test period the bronze had failed and the tests were stopped.

Images in figure 11 describe the behavior of the B2 sample:

- after 5 min of exposure an area of unsoldering appear with a low water infiltration and a significant number of small sized voids. Optical microscope analysis reveals a large number of perforations of the polymer film facilitating during the oscillations, water and air penetration. The same analysis showed the existence of small pits at the peripheral area;

- after 30 min the area of the polymer film detachment is increased and there are also large areas in which the polymer fil was removed;

- after 45 min the entire film is detached and the metallic surface has an rough aspect characteristic to the cavitation

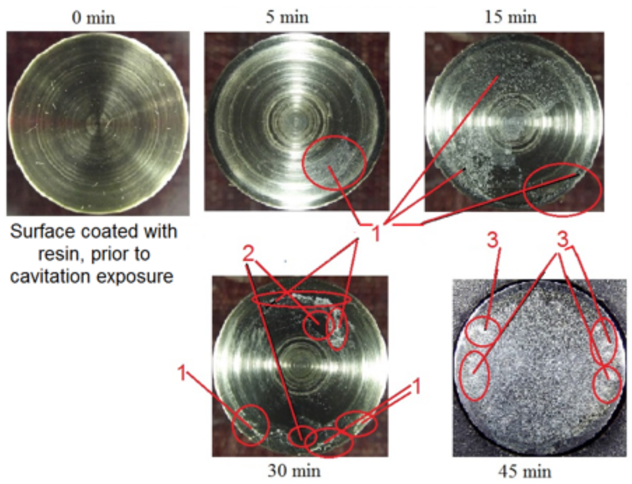


Fig.11. Behavior evolution to vibratory cavitation of the epoxy resin layers B2 deposited on the flat surface of the sample
1. detached resin film and bubbles with small dimensions; 2. the area remaining after the resin film expelled; 3.cracks/pits

erosion. This observation leads to the hypothesis that the total detachment of the polymer film took place during the first moments of the third test period (the one from 30 to 45 min). The total detachment of the polymer film allowed the attack of shockwaves directly on the metallic surface.

From the above presented analysis one observation is obvious - during the first 5 min of exposure, due to the tensions induced by the impacts with the micro-jets or due the implosion of cavitation bubbles, unsoldering of film polymer appear. The dimensions of these zones seem to be dependent both on the polymer used and film applying technique but their appearance is not depending on polymer and technique. Once the duration of exposure is increased the metal exposed area is wider, with pitting and other specific erosion effects appearing as they are known for this specific hydrodynamic phenomenon [17, 20].

From the four tested probes, regarding the behavior till 14th-15th min of exposure the two probes denoted as A ensure a better resistance to cavitation erosions.

Comparing the behavior of these modified polymers with other composite materials (thermico WC-9Co-5Cr-1Ni Ceramic Powder deposed HVOF on X2CrNiMoN22-5-3 stainless steel [19] and composite material with copper base (47.08 %Cu, 28.83 % Zn, 16.2 % Ni, 6.64 % Cr, 0.32 % Ti, 0.27 % PT, 0.27 % Co, 0.22 % Fe, 0.17 % Mo)deposed HVOF on carbon steel 270-480 W [16], developed at our laboratory) and that detached during the first 5 min of test it can be said that the modified polymers are a better solution to be used as protective surfaces on metallic samples exposed to vibratory cavitation. The research has to be continued to find the best application technique and the best films composition to obtain the best protection of the metallic surfaces. Especially the best adhesion has a great importance.

It is also important that 2 types of films (the ones for which the second layer was polymerized at environmental temperature) showed a better resistance meaning that those films are more adherent and more flexible. It is sure that the quality of such films can be improved by a fine tuning of applied parameters (the duration of keeping the metallic sample into the pre-polymer mixture, the more rigorous control of the temperature value, the increase of applied layers).

Notice: The pot with water was emptied after each test, and the water was filtered in order to retain all the polymer pieces. Thickness measurement revealed a thickness of about 75-90 µm for each sample.

Conclusions

Unsoldering of polymer films in the form of polymer sheets shows that the films are elastic and resistant but it is necessary to improve the technique to obtain a better adhesion to the metallic surface -Copper alloy in our case (bronze) -to offer a higher resistance to breaking, unsoldering and perforations under the shocks generated by the impacts with the cavitation micro-jets.

Firstly the adherence between polymer and the metallic surface (in this case Copper alloy) has to be increased because the propagation of plane waves and the repeated impacts with cavitation micro-jets the film is detached from the surface. When the thin film is cracked it allows water and air penetration in the space between polymer film and metal surface which under the effect of oscillations produce more detachments of the film, letting finally the metallic surface exposed to erosion.

The fact that A type films are more resistant than the B type ones, visible till the 15th min, show that the technique used to realize the polymer mixture can be improved and also that the presence of Scandium is better than the presence of Yttrium.

These conclusions are suggesting the continuation of research in order to find the appropriate solutions to confer for epoxy resins or polymer mixtures a better resistance to the cavitation exposure. Such solutions could offer cheap alternatives for ships propellers as well as hydraulic machines runner well protected against the effects of cavitation erosion.

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