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Advanced material fracture surfaces study using combination method of analysis and material testing

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Abstract
The paper presents a combination method of material testing and computer surface analysis applied on advanced materials. Combination method TT (Tensile Test) and SEM (Scanning Electron Microscopy) shows the importance of relieving and intimate comprehension of advanced material cracking mechanisms, with impact on the evaluation reliable level made for the lifetime of components.
Used in combination with classic destructive methods (TT), SEM method is useful for advanced material degradation evaluation and it can be applied with success to study the material/product scratching predisposition at the beginning and during the exploitation.
The method can be applied to evaluate actual state of components made from advanced materials and also for micromechanical behavior of these components under mechanical loading. In this way we have the necessary information about the exploitation behavior of the component/material, in the presence of defects, before these appear and develop failures.
The paper presents the results obtained on thermoplastics materials used for transportation of under pressure fluids and to analyze the electrochemically deposited layers of MoO and CuInS.
Keywords: SEM, advanced materials, combinative / hybrid tests, material evaluation actual state, residual lifetime

1. General information about the methods used for advanced material testing

A very important direction in advanced material domain is to produce advanced materials and/or to find a way to improve the properties of materials used in this moment. Before initiate a new material we must test it to see the lifetime and its properties [1].

For material testing, without damages, non- destructive methods can be used. The principal purpose of the NDT methods is to evaluate the performance of the component during the lifetime. These methods are used: when the component is produced, when putting into operation, after a given functioning time (a short time), to estimate the component residual lifetime.

For integrity analyse are used over 50 examination methods, but for better results are used the combined or hybrid methods.

For advanced material defects hybrid method are especially used which combine classic mechanical testing and non- destructive methods (acoustics emission, thermography, laser extensometry). Appling in the same time all these methods we will obtain quantitative information about morphological properties [2, 3].

In the case of composite materials, especially „sandwich" structure type, active and passive thermography with infrared radiation are used [4].

Using active thermography exfoliation can be detected and defined [5] between the layers in different testing stages and the passive thermography can locate the fracture initiation.

In this paper is presented the SEM (Scanning Electron Microscope) examination method, as a complementary method for classic mechanical testing (MT).

2. Working procedure

2.1. Experimental material

For experimental work thermoplastics and composite materials were used:

First material: polyethylene pipes PE 80 with standard nominal diameter ϕ 32 $\times$ 3 mm, that were pre-cracked by the LSI Method [1] and after that were tested at the internal pressure on a constant temperature to the creep, to evaluate the notching sensibility of material.

Testing parameters used:

- Pressure: 10 bar
- Temperature: 80˚C
- Testing medium: water

Second material: MoO_{3-x} and CuInS layers. These layers were electrochemically deposited and after that heat treated to 450^0 C.

2.2. Examination method

To study micromechanical properties of materials the SEM method (Scanning Electron Microscop) was used.

This method can be applied using an electron microscope able to produce high resolution image. Using scanning technique three dimensional images of fracture surfaces can be obtained.

Surfaces must be perfectly cleaned, without grease and dry, without dust and other impurities. It can be accomplished using a solvent and after that warm air – drying.

In the case of non conductive materials, in order to examine the fracture character, after degreasing samples must be prepared. High vacuum evaporation is one of the more used ways of applying thin-metal and carbon films as coating layers on specimens. The noble metals and their alloys are highly conductive and are suitable as coating materials for most samples to be imaged using secondary electrons. The same process is used for metallographic replica study [6].

The main function of this layer must be to increase the surface electrical conductivity of the sample with the additional attributes of increasing the thermal conductivity. The first condition to analyze a sample with SEM to be conductive wire is fulfilling. This method has good results and is used for the examination of the structures within ISIM Timişoara's laboratory.

2.3. Experimental equipment

To study the fracture surfaces the scanning electron microscope BS 434 A Tesla and a computer to store images delivered by microscope (figure 1).

Figure 1. Experimental Setup

Technical specifications:

- Magnification range on screen: 15 – 50.000 x
- Working distance between 8 – 50 mm
- Maximum specimen diameters and height are 85 mm and 22 mm respectively

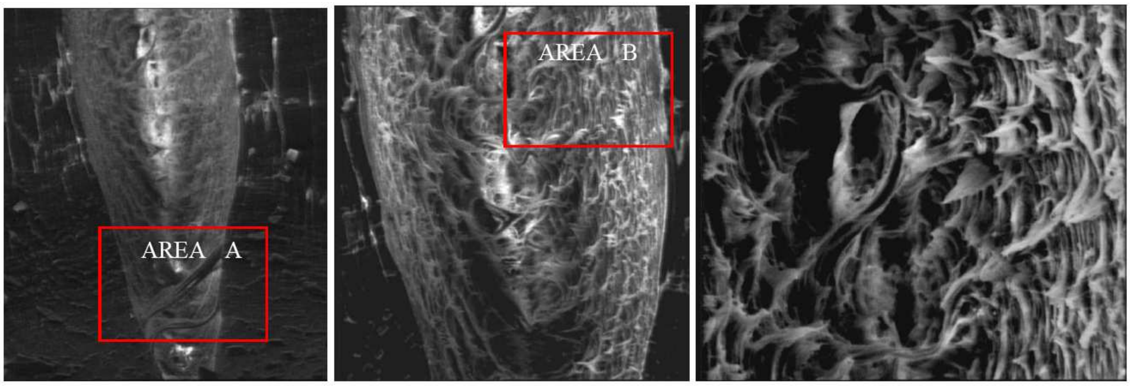
3. Experimental results

Experimental results obtained on tested advanced material within ISIM Timişoara's laboratory. Results are images of fracture surfaces obtained using the SEM method.

Figure 2 presents the experimental results of first materials using the computer electron microscope method SEM. There are fracture areas results from testing at the internal pressure.

The analyse shows a material fibrous texture, and parallel fibres with the pipe axis. Symmetrically developed fracture compared with initial notching axis has an elliptic form with two centres representing local stress concentrators.

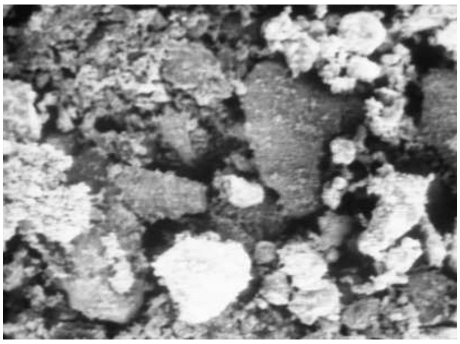
Both plastic deformation of fibres (A – initiation area) in the vicinity of the two centres and fibre cracks (B – central area) were evinced.



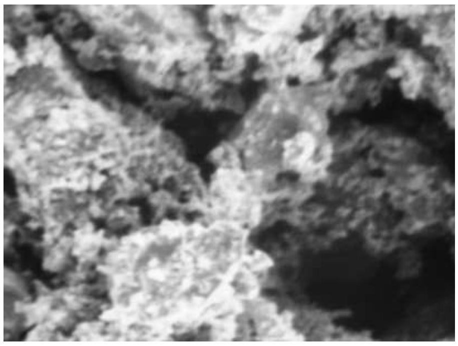
a) 120X b) 240X c) 480 X – B area detail

Figure 2. Polyethylene pipe PE 80 fracture surface image obtain using SEM method testing at the internal pressure on a constant temperature to the creep, after 8500 testing hours

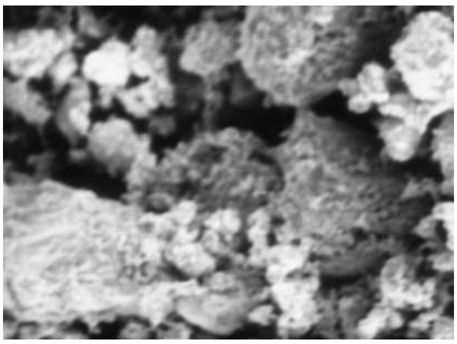
Figure 3 shows $CuInS_2$ crystal edges. In the sample there are also some Cu and In alloys. Because the sample is not covered with Pt or Au we can identify few lighting areas especially for metallic alloy. In this purpose backscattered electrons detector was used.



a) (65 µm, x 240)



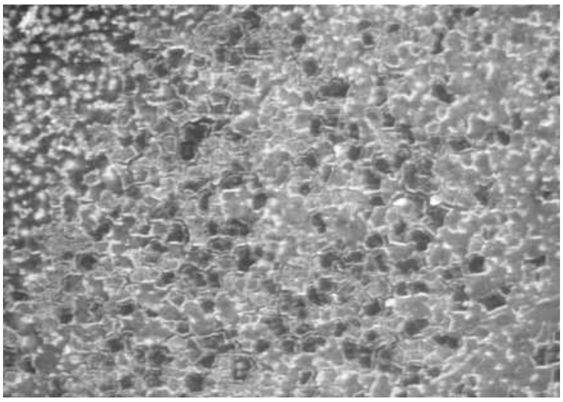
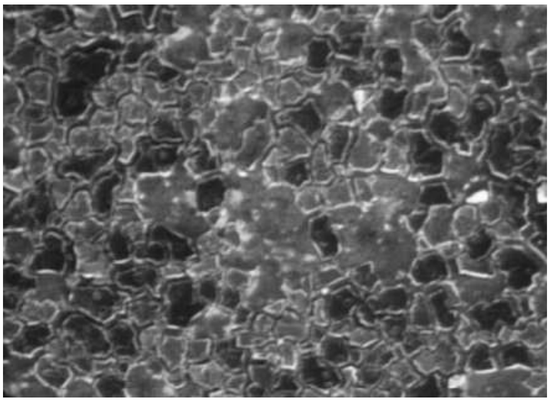
b) (65 µm, x 480)



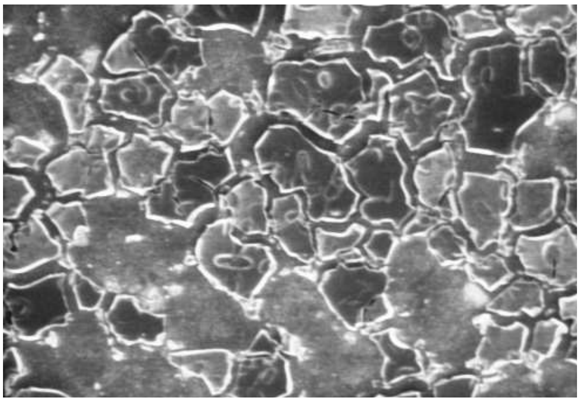
c) (65 µm, x 750)

Figure 3. CuInS powder grains

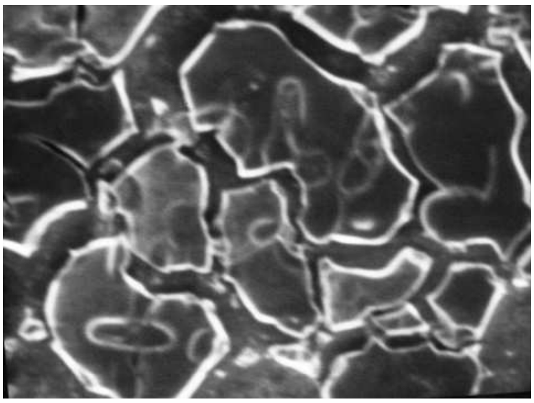
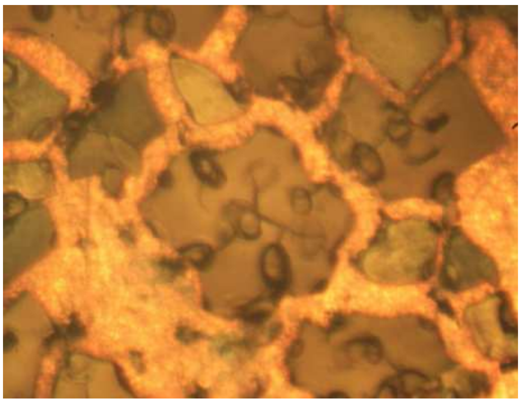
In figure 4 oval formations can be observed. In the growth process of the oxygen layer in the oval formations appeared hydrogen bubbles. Where bubbles were, holes (craters) remained, after the bubbles disappeared. The lifetime of the bubbles was about few seconds or even minutes. Because the water was eliminated very quickly and dilatation coefficients between the cooper layer and oxygen layer was different, layers presented crevices.

a) (65 µm, x 24)   b) (65 µm, x 48)



c) (65 µm, x 120)

d) (65 µm, x 240)   e)magnified 120x(optical microscope)

Figure 4. MoO layers

4. Concusions

4.1. Today the global tendency is to use the combined or hybrid testing methods to study the properties of composite and thermoplastics materials.

4.2. The SEM method is an operative examination method that can be successfully applied on advanced materials components, in combination with different testing mechanics methods.

4.3. Using the MT-SEM method very important information is obtained about the initial state of the materials, information that can be used to evaluate the behaviour of the material during the testing process.

4.4. The MT-SEM method is very useful to evaluate the notching sensibility

4.5. Mo oxides are used as precursors for Mo sulphurs, in solar cells, as catalysts in organic chemistry, in the construction of accumulators etc.

4.6. CuInS layers are used in solar cells.

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