

## FRACTOGRAPHIC EVALUATION OF THE METALLIC MATERIALS FOR MEDICAL APPLICATIONS

Brandusa GHIBAN<sup>1</sup>

*The manner of studying of the fracture modes could be done through fractography. Fractography is the study of fracture surface morphologies and it gives an insight into damage and failure mechanisms, underpinning the development of physically-based failure criteria. In composites research it provides a crucial link between predictive models and experimental observations. Fractographic methods are routinely used to determine the cause of failure in all engineering structures, especially in product failure and the practice of forensic engineering or failure analysis. In material science research, fractography is used to develop and evaluate theoretical models of crack growth behavior. One of the aims of fractographic examination is to determine the cause of failure by studying the characteristics of a fracture surface. Different types of crack growth produce characteristic features on the surface, which can be used to help identify the failure mode. The overall pattern of cracking can be more important than a single crack, however, especially in the case of brittle behavior materials. Initial fractographic examination is commonly carried out on a macro scale utilizing low power optical microscopy and oblique lighting techniques to identify the extent of cracking, possible modes and likely origins. Optical microscopy at low magnification or the so-called macrophotography (and sometimes when a stereomicroscope is used—stereomicroscopy) are often enough to pinpoint the nature of the failure and the causes of crack initiation and growth if the loading pattern is known. When it is needed to identify the nature of failure, an analysis at high magnification is required and scanning electron microscopy (SEM) seems to be the best choice. The problem of fracture behavior of biometallic materials is a real one, being well and repeatedly presented in literature. The human body is not an environment that one would consider hospitable for an implanted metal alloy: a highly oxygenated saline electrolyte at a pH of around 7.4 and a temperature around 37°C. While it is well known that chloride solutions are among the most aggressive and corrosive to metals, the ionic composition and protein concentration in body fluids complicate the nascent understanding of biomedical corrosion even further. Variations in alloy compositions can lead to subtle differences in mechanical, physical, or electrochemical properties. However, these differences are minor compared with the potential variability caused by differences in fabrication methodology, heat treatment, cold working, and surface finishing, where surface treatments are particularly important for corrosion and wear properties. The aim of this presentation, therefore, is to summarize the different types of metals and alloys used as biomaterials, the corrosion of metals in the human body, and different failure damages of implant metallic materials.*

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<sup>1</sup> Prof., Dept.of Metallic Materials Science, Physical Metallurgy, University POLITEHNICA of Bucharest, Romania, e-mail: ghibanbrandusa@yahoo.com

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## 1. Introduction

Implant alloys are typically derived from three materials systems: stainless steels, cobalt-chromium based alloys and titanium alloys [1]. The question “Does corrosion of a metallic implant cause a clinically relevant problem?” is one that probably only an electrochemist or materials engineer will ask when confronted with the prospect of having a metal device implanted into his or her body. While numerous issues may arise with the implant following surgery, one of the most fundamentally important is the interaction between the surrounding physiological environment and the surface of the implant itself. This interaction can lead to either the failure of the implant to function as it was intended, or have an adverse effect on the patient Metal Corrosion in the Human Body: The Ultimate Bio-Corrosion Scenario by Douglas C. Hansen resulting in the rejection of the implant by the surrounding tissue, or both [2]. In either case, explanation of the device is usually required to correct the situation.

The human body is not an environment that one would consider hospitable for an implanted metal alloy: a highly oxygenated saline electrolyte at a pH of around 7.4 and a temperature around 37°C. While it is well known that chloride solutions are among the most aggressive and corrosive to metals, the ionic composition and protein concentration in body fluids complicate the nascent understanding of biomedical corrosion even further. Variations in alloy compositions can lead to subtle differences in mechanical, physical, or electrochemical properties. However, these differences are minor compared with the potential variability caused by differences in fabrication methodology, heat treatment, cold working, and surface finishing, where surface treatments are particularly important for corrosion and wear properties. Since metals are inherently susceptible to corrosion, implants are routinely pre-passivated prior to final packaging using an acid bath or some other electrochemical anodizing process (titanium alloys),[4] or an electropolishing method (stainless steel and cobalt alloys) [5].

Fracture is a form of failure where the material separates in pieces due to stress, at temperatures below the melting point. The fracture is termed ductile or brittle depending on whether the elongation is large or small. There are several types of failure for a given metallic material used as biomaterial, as is illustrated in Fig. 1, such as Foreign object damage (FOD), Fatigue failures, Stress corrosion cracking, Corrosion and passivation, fretting, creep, erosion, cavitation [10-11].

On the other hand, there are specific requirements for the implant / prosthesis, as is given in Fig. 2, such as compatibility, mechanical properties and manufacturing.

The aim of this article, therefore, is to illustrate how may appear failure on two types of implants, one used for self - locking and the other one for hip revision.

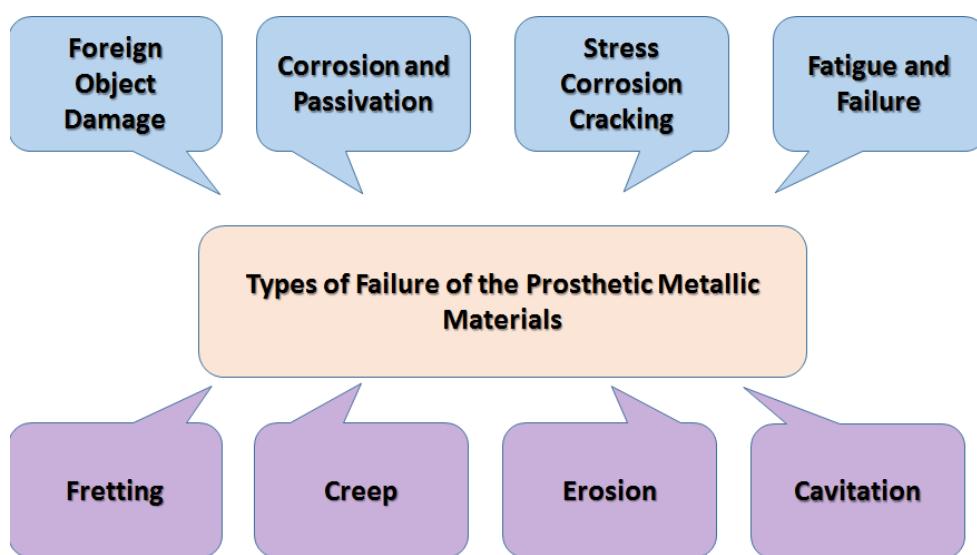


Fig. 1- Main types of failure of the prosthetic metallic materials

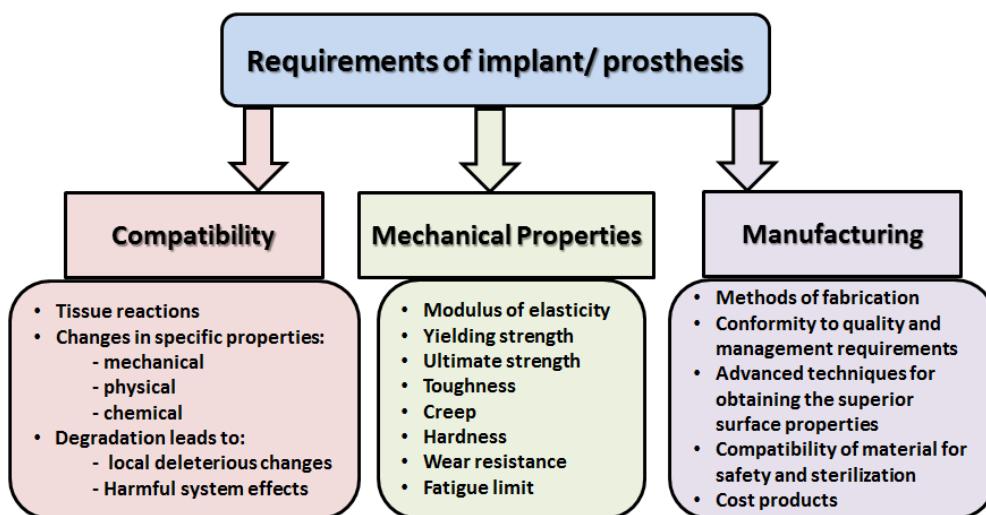


Fig. 2- Requirements of implant / prosthesis

## 2. Materials and Methods

Present paper investigates two case studies: one concerning self locking plate, and the other one a hip implant. Self-locking plate was used for reduction and fixation to a tibial pilon fracture for patient aged 30 years. After eight months the patient returns for the ablation of metallic implant. Clinical and strengthen the fracture callus is found radiologic hypertrophic and degradation of the plaque was observed. The second case study was conducted on a sample taken from the NC straightening of hip fracture patient's rod, the age of 82 years. The macroscopic aspects for self locking plate is given in Fig. 3, and for hip implant in Fig. 4. In order to establish the causes that led to the breaking of implantable materials, the following investigations were made: determination of chemical composition using spectral analysis, macro-structural analysis using the stereomicroscopy, microstructural analysis using optical microscopy, fractographic analysis using both stereomicroscopy and scanning electron microscopy. Macrostructural analysis was performed on a stereo microscope Olympus type SZX7 equipped with image processing software Quickphoto Micro 2.2. Scanning electron microscopy was made on a Philips microscope. Optical microscopy analysis of the metallic samples obtained from self-locking plate was performed with a Reichert type microscope. The metallographic analysis was made on the optical microscope, which was used coupled to an image analyser using Image Pro software.

## 3. Results and Discussion

The chemical composition for the materials of the implants in this paper was determined by spectral quantitative analysis, as is shown in table 1. Analysing the results, we could confirm that the metallic biomaterial used is pure titanium grade 4 for the self locking plate and, giving it notes that limits carbon and nitrogen contents are easily overcome [10, 11], and for the hip revision is Ti6Al4V.

### 3.2. Fractographic analysis for case study 1

Fractographic analysis made by scanning electron microscopy (Fig. 5) revealed that self-locking plate cracked in an area with high purity embedding. Breaking looks brittle by cleavage transgranular with sharp facets. There were also highlights and intergranular cracks between planes of the cleavage side. It should be noted that after tearing of the two components of self locking plate underwent relative friction that led to chamfering breaking peaks. The analysis

shows that the extraction time to rupture and self-locking plate there was a relatively long period in which the friction surfaces chamfering and breaking.

Table 1

## Chemical composition of the materials investigated in this paper

Material	Chemical composition, % wt.								
	N	C	H	Fe	O	Mo	Al	V	Ti
Samples obtained from retrieved self-locking plate	0.055	0.11	0.010	0.25	0.65	-	-	-	Rest
Sample obtained after hip revision	0.04	0.06	0.010	0.28	0.15	1.52	6.25	4.13	Rest
ISO standard specifications for pure Ti grade 4	Max. 0.05	Max. 0.10	Max. 0.0125	Max 0.3	Max 0.45	-	-	-	rest
ISO 5832/3	Max. 0.05	Max. 0.08	Max. 0.0125	Max 0.3	Max 0.02	Max 2	5.5-6.75	3.5-4.5	rest



Fig. 3- Different macroscopic aspects of the self-locking plate after stereomicroscopic analysis: a- right side view, b- back side view



Fig. 4- Macroscopic aspects of the hip rod with the evidence of the fracture head (a) and changing in color at the zone between mounting holes (b)

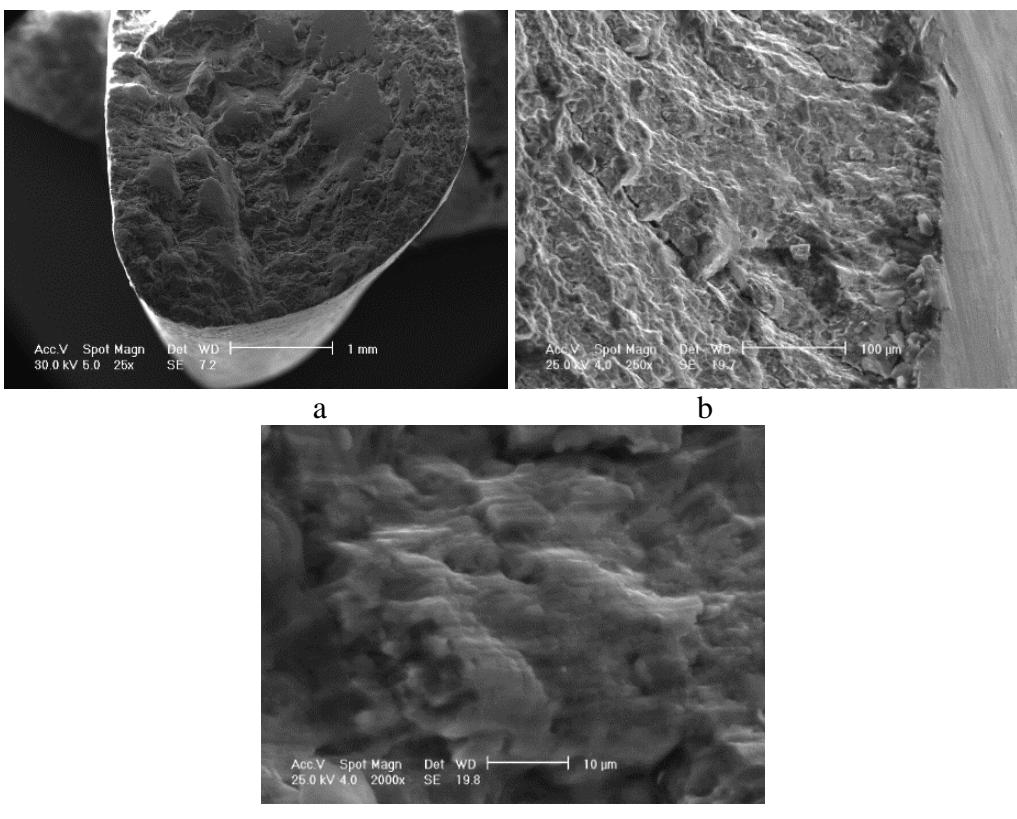
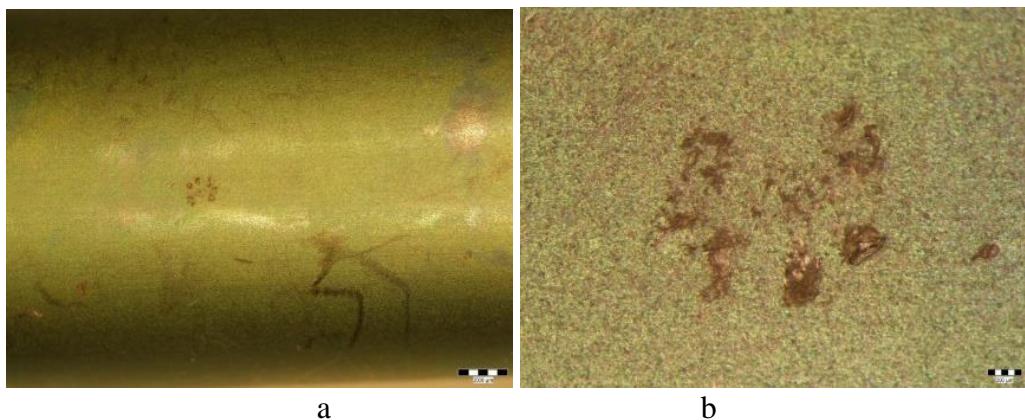


Fig. 5- Fractographic images at SEM in transversal cross section of the retrieved self-locking plate at different magnifications

### 3.3 Fractographic analysis for case study 2

The investigated rod used for hip revision has the dimensions  $\Phi 10\text{mm} \times 125^\circ$ . Note that the investigated area comes after thread, there are traces of interaction with the bone, such as color changes (Fig. 6a), or the presence of a surface defect (Fig. 6b). We have the observation of two comcatity defects on the rod, either on the external surface (such as macroporozities (Fig. 6a and Fig. 6b), or dimple on internal surface (as is illustrated in Fig. 6c). On the rod under fractographic stereo macroscopic review a number of issues were highlighted, as can be seen in Fig. 7. Both sides breaking the impression of fatigue breaks, with clear evidence of the three specific areas of strength: the initiation of fracture (lateral ends of Fig. 7a and Fig. 7c); propagation area, beach marks, with concentric arcs appearance (as demonstrated in Fig. 7b and Fig. 7d), and the suddenly final rupture (having the appearance chamfered).

Fractographic analysis made on scanning electron microscope highlighted the character of fatigue breaks, with all specific zone: initiation (Fig. 8a), propagation zone (Fig. 8b) and final fracture (Fig. 8c). The fractographic images from Fig. 8 highlights aspects of fatigue fracture in the final sudden fracture, respectively the opposite end initiate fatigue fracture were evidenced. Breaking character is fragile, by cleavage with numerous fine secondary cracks, and voids, probably between the two phases type  $\alpha$  and  $\beta$  present of Ti6Al4V titanium alloy rod belonging hip investigated.





c

Fig. 6- Macroscopic aspects of the hip rod, with the evidence of a compactity defect on external surface (a,b), and a dimple defect on internal surface (c)

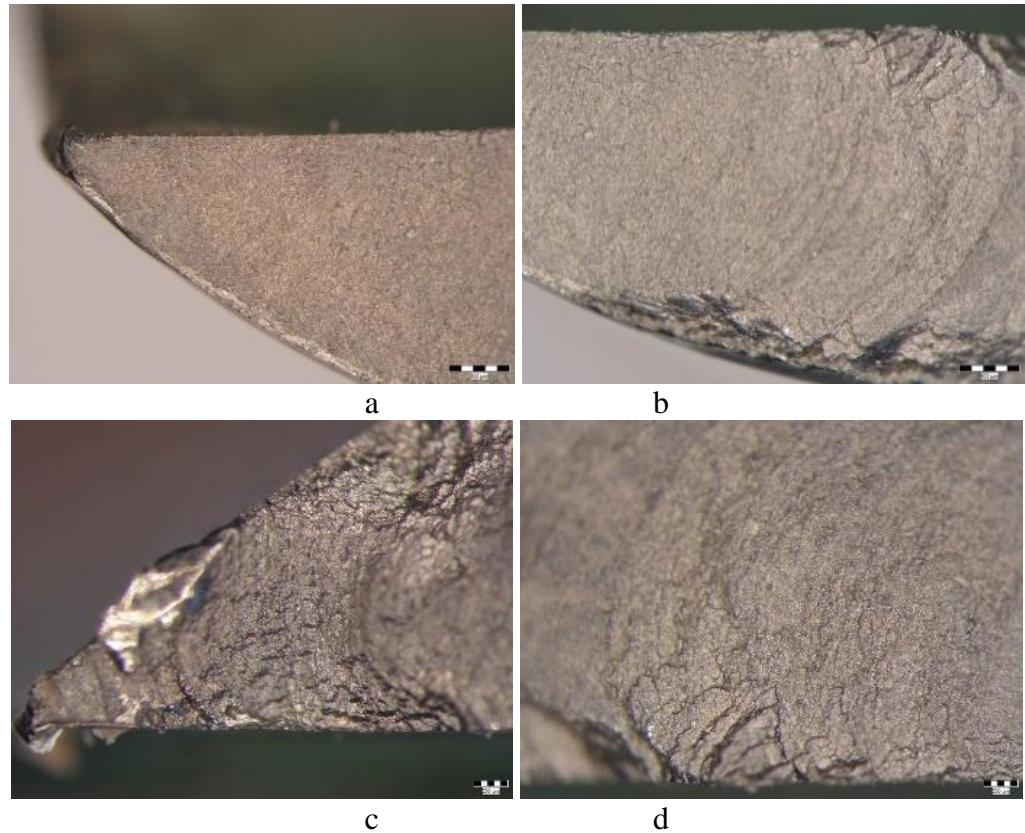


Fig. 7– Fractographic aspects of the hip rod  
(a,b lower part band), (c,d upper part band)

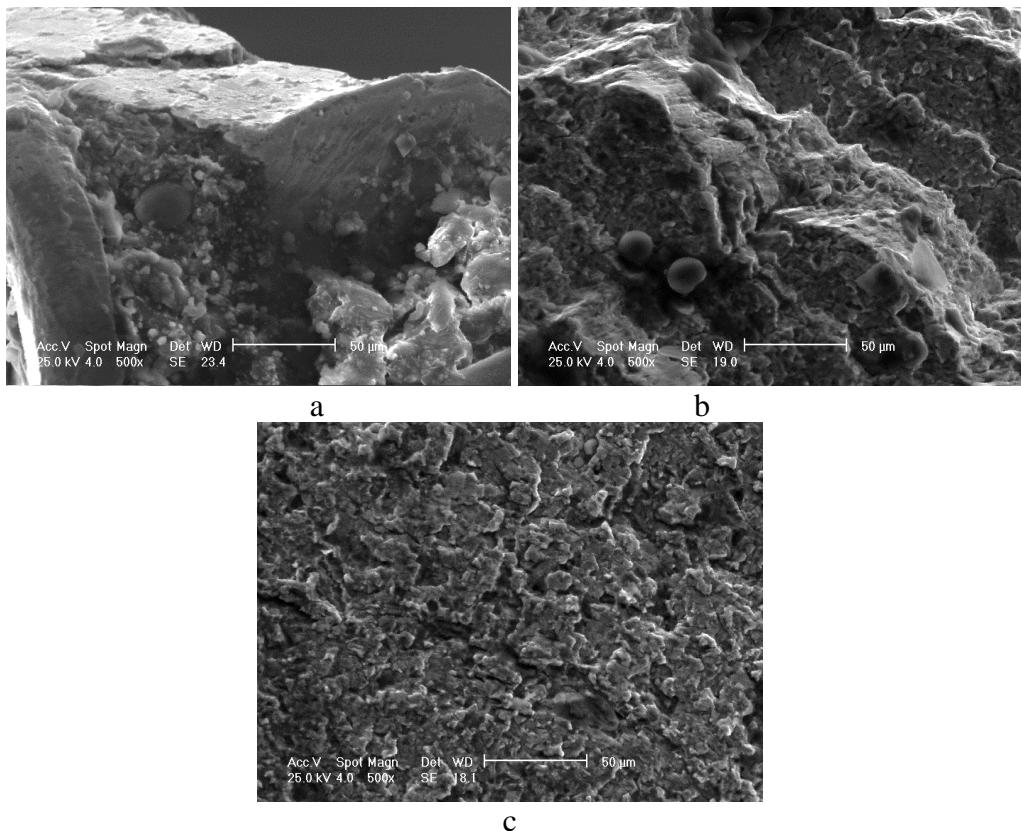


Fig. 8- Fractographic aspects of the rod for hip revision:  
a- initiation zone, b- propagation zone, c- final fracture zone

#### 4. Conclusions

The present paper displays the results concerning fracture behavior of two study cases, respectively one used for self-locking and the other one for hip revision.

**Concerning the first case study,** complete analysis of the fracture surfaces of the plate-locking led to the final conclusion that the metallic biomaterial which has been made self-locking plate, respectively pure titanium grade 4, has an inadequate chemical purity in term of non-metallic impurities, which has led to fracture within an area of non-homogeneous structure. Breaking looks brittle by transgranular cleavage with sharp facets. There were also highlights and secondary intergranular cracks between cleavage planes. Failure analysis of different metallic implants used in traumatology appear to be very useful for biomaterials scientist but especially to the manufacturers of orthopaedic implants. They must pay attention to the metallic biomaterials microstructure not just to the chemical composition and to the processing technologies that could

modify the metallic biomaterials structure. This kind of problems could generate not just potentials problems related to the biomechanical functionality but also the biocompatibility problems.

**Concerning the second case study** the rod material is made of titanium alloy type Ti6Al4V, falling trade requirements of ISO 5832. The rod was broken after one year of implantation, having the dimensions as  $\Phi 10\text{mm} \times 125^\circ$ . Investigation of the area after thread, there are traces of interaction with the bone, such as color change and presence of a surface defect. On macroscopic stereo rod under review were highlighted a number of issues fractographic. Both sides breaking the impression of fatigue breaks, with clear evidence of breaking the three specific areas: the initiation of the tearing (the side ends); the propagation, with beach marks line, appearance concentric arcs, and the final sudden fracture (the remainder heads having beveled appearance). fractographic aspects highlighted the character of fatigue breaks, but compactness of defects, both on the outer surface and in the internal surface of the stem lead to the general conclusion that the material is not as described product, having problems with the technological processes before implantation.

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