Available online at www.sciencedirect.com

ScienceDirect

Procedia Structural Integrity 28 (2020) 45–52

Structural Integrity Procedia

www.elsevier.com/locate/procedia

1st Virtual European Conference on Fracture

Application of the Theory of the Critical Distances based methodology for the analysis of Environmentally Assisted Cracking processes in biomaterials

P. González[a], S. Cicero[a,*] , M. Peron[b], B. Arroyo[a], J.A. Álvarez[a], F. Berto[b]

[a]LADICIM - University of Cantabria, E.T.S. de Ingeneros de Caminos, Canales y Puertos, Avda. Los Castros 44, 39005, Santander, Spain
[b]Dep. Industrial & Mechanical Eng., Norwegian University of Science & Technology, Richard Birkelands vei 2b, 7034, Trondheim, Norway

---

Abstract

The complex interaction between physiological stresses and corrosive human fluids can lead to the premature failure of metallic biomaterials due to the development of Environmental Assisted Cracking (EAC) processes. In this paper, the EAC phenomenon is analysed through a Theory of Critical Distances based methodology, which has been validated in other materials and aggressive environments, and the apparent crack propagation threshold in notched conditions is estimated. Notch-like defects, which are frequently found in aggressive environments, may present higher values of crack propagation thresholds than those exhibited in cracked components. The knowledge of this higher material performance makes it possible to address the problem avoiding oversizing or unnecessary replacements in biomaterials, which leads to an improvement in the quality of life of the people carrying these materials.

In this study, the susceptibility of AZ31 magnesium alloy to EAC and the evolution of the apparent crack propagation threshold have been analysed. The aggressive environment used was Simulated Body Fluid (SBF). The main conclusion is that the Theory of Critical Distances predicts the behaviour of this biomaterial in notched conditions and subjected to the aggressive environment being studied.

Keywords: Theory of Critical Distances; Environmental Assisted Cracking; Biomaterials; Magnesioum alloy.

---

* Corresponding author. Tel.: +34-942-200-017
E-mail address: ciceros@unican.es

10.1016/j.prostr.2020.11.130

1. Introduction

In the last few decades, life expectancy, which has been gradually increasing, has led to a growth in surgeries for the implantation of medical devices (Ginebra et al. (2006)). The most important surgery is the orthopedic one, which also has the highest annual growth rate (Long and Rack (1998), Long (2008)). The most commonly used materials in this area are metals (i.e. stainless steel, titanium and cobalt-chromium alloys, Chen and Thouas (2015)) that have a permanent-implant character. The use of these materials is motivated by their good mechanical properties and corrosion resistance, which means that they are frequently used for bone healing and reparation of damaged tissues (Albrektsson et al. (1981), Hanawa (2010)). The fundamental aspects to be analysed in permanent implants are the difference in the modulus of elasticity between these materials and the bones, while the appearance of long-term complications should be considered. Some implants have to be removed once the healing process has ended (Engh and Bobyn (1988), Jacobs et al. (1998)). However, the surgeries necessary to remove them increase the healthcare costs and the emotional stress of the patient. For this reason, biodegradable materials are currently being studied, Magnesium (Mg) and its alloys being considered good candidates for biomedical applications (Li and Zheng (2013), Peron et al. (2017), Hänzi et al (2009)). Despite their highly attractive properties, Mg and its alloys have not yet been used as implant materials due to their high corrosion rates in the physiological environment, which can lead to a loss of mechanical integrity and hydrogen diffusion which may be too fast for the bone tissue to accommodate. In addition, in orthopedic applications, the implant materials must have a good resistance to Environmental Assisted Cracking (EAC), since the mechanical loads of the human body and its movements are combined synergistically with aggressive environments such as human fluids.

Environmental Assisted Cracking (EAC) has been identified as the cause of failure of several traditional implants (Jafari et al. (2015), Teoh (2000), Akahori et al. (2000)). This phenomenon is particularly dangerous because it leads to fast, sudden and catastrophic failures under conditions of mechanical loads lower than the yield stress of the material.

In this context, it has been proved that Mg and its alloys are susceptible to the aforementioned EAC phenomena. Therefore it is important to develop implants that guarantee a good resistance both to EAC as well as to the mechanical loads of the human body (Jafari et al. (2017), Jafari et al. (2018)). However, most studies have focused on improving the electrochemical properties of Mg and its alloys, while research on the behaviour of Mg against EAC is very limited. In fact, different procedures to improve its corrosion resistance have been established over the last few years, from alloying to surface modification techniques; only a few of these have been evaluated considering their susceptibility effects when facing EAC (Mohajernia et al. (2018)).

These studies of the EAC in the field of human health need to guarantee the integrity of the components precisely, since very conservative results will lead to a greater health expenditure on surgical procedures and a higher personal cost for the patients.

Nomenclature

Mg	Magnesium
EAC	Environmental Assisted Cracking
TCD	Theory of Critical Distances
L	Critical Length
K_{mat}	Fracture Toughness of the Material
σ_0	Inherent Stress
PM	Point Method
LM	Line Method
K^N_{mat}	Apparent Fracture Toughness
ρ	Notch radius
SBF	Simulated Body Fluid
CSE	Calomel Saturated Electrode
K^N_{IEAC}	Apparent crack propagation threshold in an aggressive environment for notched components
K_{IEAC}	Crack propagation threshold in an aggressive environment

P_Q Load for the beginning of the crack propagation
a Crack Length

In structural integrity assessments of the components used as prostheses the appearance of defects is a common finding. If these defects have a certain degree of rounding or blunting on their edge, they should be considered as notches rather than cracks. The perception of them as cracks will lead to imprecise and overconservative results of their resistance to cracking in the environmental conditions studied. This fact shows the need to implement analysis methodologies that take into account the real behaviour of the notched components.

The Theory of Critical Distances (TCD) has been used for the analysis of the EAC in notched components in aggressive environments typical of the oil and gas extraction and transportation industry, or the power generation industry (González et al. (2019a), González et al. (2019b)). In this work, the application of the TCD methodology will be carried out to study its phenomenon in other aggressive materials and environments, as is the interaction of Mg and corrosive fluids in the human body.

2. Theory of Critical Distances

The Theory of Critical Distances (TCD) groups a set of methodologies that employ the mechanics of continuous media together with a characteristic parameter of the material named critical length, L, to predict the behaviour of components in the presence of notches, or other stress concentrators different from cracks, for fracture and fatigue assessments (Taylor (2007)). TCD was first postulated in the mid-twentieth century (Neuber (1958), Peterson (1959)) but it has been during the last 20 years that this methodology has advanced significantly to provide answers to different engineering problems (e.g., Susmel and Taylor (2008), Susmel and Taylor (2010), Cicero et al. (2012)). The aforementioned length parameter, L, is the critical distance and, for fracture analysis follows equation (1):

$$L = \frac{1}{\pi}\left(\frac{K_{mat}}{\sigma_0}\right)^2 \quad (1)$$

where K_{mat} is the fracture toughness of the material and σ_0 is the inherent stress, which is usually greater than the elastic limit of the material but requires calibration.

The two simplest and most used methodologies of TCD are the Point Method (PM) and the Line Method (LM); LM is the one employed in this work. The Line Method establishes as a failure criteria, equation 2, that the average stress from the notch tip along a length equal to 2L reaches the value of σ_0 (Figure 1):

$$\frac{1}{2L}\int_0^{2L} \sigma(r)dr = \sigma_0 \quad (2)$$

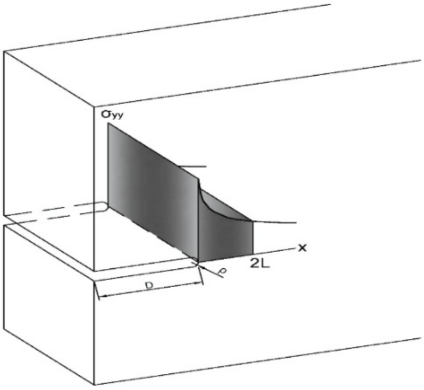
This method is able to predict the apparent fracture toughness (K^N_{mat}) for U-shaped notches when combined with the linear-elastic stress distribution in the notch tip proposed by Creager-Paris (1967), which coincides with the stress distribution at imaginary crack tip displaced a distance equal to ρ/2 along the x-axis, located in the bisector plane of the notch and originated at its tip, ρ being the radius of the notch (equation (3)).

$$\sigma(r) = \frac{K}{\sqrt{\pi}}\frac{2(r+\rho)}{(2r+\rho)^{\frac{3}{2}}} \quad (3)$$

Equation (4) then allows K^N_{mat} to be predicted by the PM in fracture assessments as a function of the fracture toughness of the material, K_{mat}, the notch radius, ρ, and the critical distance, L.

$$K^N_{mat} = K_{mat}\sqrt{1+\frac{\rho}{4L}} \quad (4)$$

Fig. 1. Definition of the Line Method.



3. Materials and Methods

3.1. Material and Aggressive Environment employed

The material employed in this work was the AZ31 Magnesium alloy, whose microstructure as received is shown in Figure 2. It consists of a homogeneous matrix α with a grain size of 13.2 ± 8 µm (Peron et al. (2019)). A tensile test was performed in order to obtain the mechanical properties of the material, together with the Stress-Strain graph, that are shown Table 1 and Figure 3, respectively:

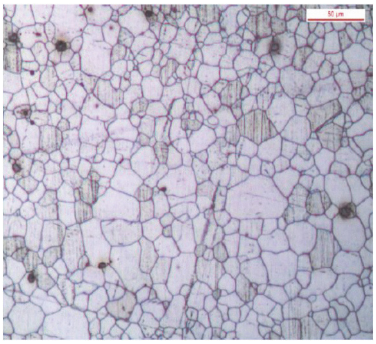


Fig. 2. Microstructure of AZ31 alloy (Peron et al (2019)).

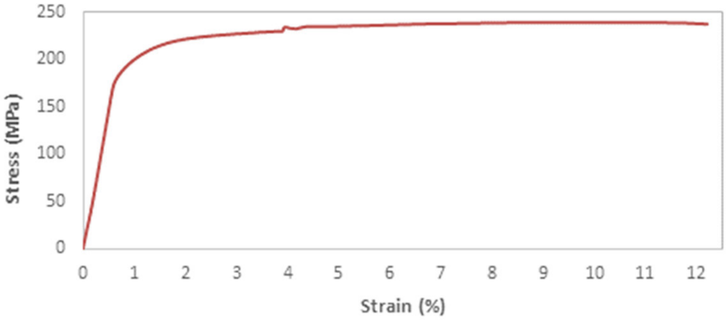


Fig. 3. Tensile curve of AZ31 alloy.

Table 1. Mechanical properties of AZ31 alloy.

σ_y *(MPa)*	σ_u *(MPa)*	E *(MPa)*	e_{max} *(%)*	K_{mat} *(MPa·m$^{1/2}$)*
190.30	238.80	31000	12.21	23.75

The aggressive environment used corresponds to a solution that simulates the human fluid, known as SBF (Simulated Body Fluid) prepared by the method of Kokubo Takadama (2006), that requires the following reagents in the given quantities for 1000 ml of dissolution (Table 2):

Table 2. Reagents for 1000 ml of SBF preparation

NaCl	$NaHCO_3$	KCl	$K_2HPO_4 \cdot 3H_2O$	$MgCl_2 \cdot 6H_2O$	$CaCl_2$	Na_2SO_4	$((HOCH_2)3CNH_2)$ (Tris)	1M-HCl	4, 7, 9 pH pattern
8.035g	0.355g	0.225g	0.231g	0.311g	0.292g	0.072g	6.118g	39ml	-

It must be ensured that the solution remains colourless, transparent and without any sediment at the bottom of the containers at any time. The temperature and pH must remain between 36 ± 1.5 °C and 7.40 respectively.

In order to analyse the phenomenon of EAC, the specimens employed in this work have been continuously cathodically charged to a constant potential of -200 mV vs Calomel Saturated Electrode, SCE; for this purpose a potentiostat was used. The schematic representation of the test setup is shown in Figure 4.

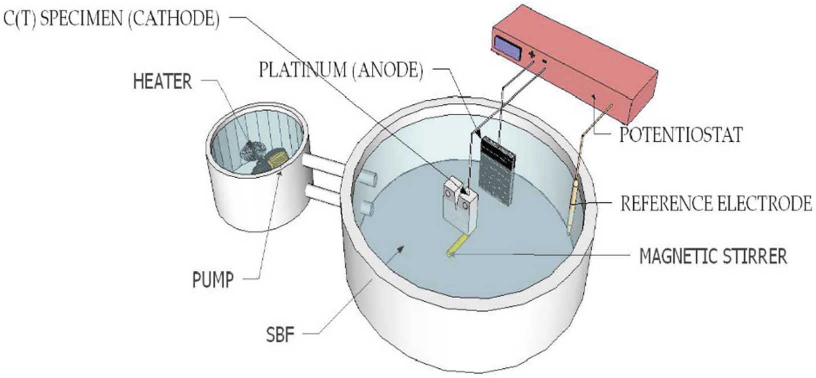


Fig. 4. Schematic of the experimental setup.

3.2. Methodology for EAC analysis based on the TCD

In this work, the Point Method has been used through the expressions proposed for the evaluation of the EAC (González et al. (2019a), Gonzalez et al. (2019b)), where the apparent crack propagation threshold in an aggressive environment for notched components, K^N_{IEAC}, is linked to the crack propagation threshold in aggressive environment, K_{IEAC}, the notch radius, ρ, and critical distance, L, by equation (5):

$$K^N_{IEAC} = K_{IEAC}\sqrt{1+\frac{\rho}{4L}} \tag{5}$$

Therefore, it is necessary to determine the crack propagation threshold in an aggressive environment (K_{IEAC}), by means of precracked specimens and the calibration of the parameter L (critical length) to be able to estimate K^N_{IEAC} for any possible notch radius.

The tests for the determination of both K_{IEAC} and K^N_{IEAC} will be carried out by means of precracked 25mm thick C(T) specimens with different notch radii, ρ. The test were performed at a rate of $6 \cdot 10^{-8}$ m/s in an horizontal slow rate testing machine while the applied load and the COD were continuously recorded.

The ISO 7539 standard (2015) allows the calculation of the crack propagation threshold in an aggressive environment from the load for the beginning of the crack propagation, P_Q, in EAC processes. The expression used to determine this parameter is the following one:

$$K_{IEAC} = \frac{P_Q}{(BB_N W)^{1/2}} f\left(\frac{a}{W}\right) \tag{6}$$

where B, B_N and W are geometric parameters (thickness, net thickness and width) of the C(T) specimen used and f (a/w) is a geometric factor that depends on the crack length, a, whose equation for C(T) specimen is:

$$f\left(\frac{a}{W}\right) = \frac{\left[\left(2+\frac{a}{W}\right)\left(0.886+4.64\frac{a}{W}-13.32\left(\frac{a}{W}\right)^2+14.72\left(\frac{a}{W}\right)^3-5.6\left(\frac{a}{W}\right)^4\right)\right]}{\left(1-\frac{a}{W}\right)^{3/2}} \tag{7}$$

Figure 5 shows the experimental set-up performed for the slow strain rate tests carried out.

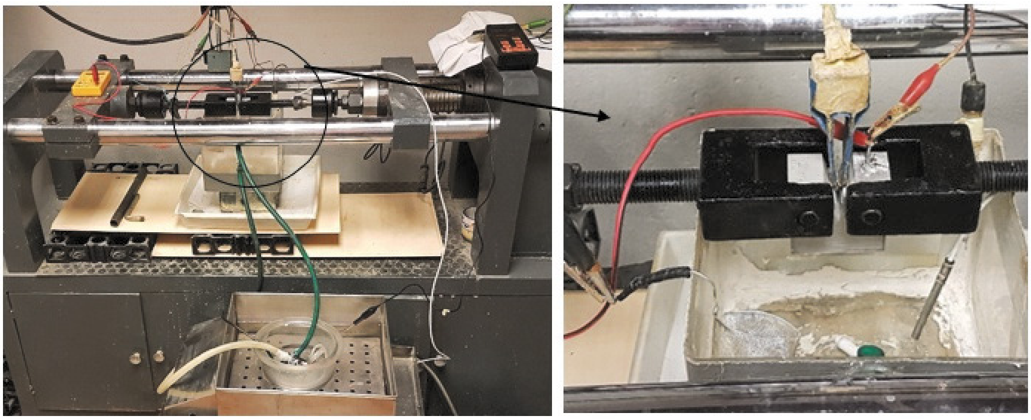


Fig. 5. Experimental set-up performed for the slow strain rate tests carried out

As recommended by the standard ISO 7539, the tests must ensure that the crack or notch is submerged in the solution during its execution and, as previously mentioned, the temperature of the solution must remain in the range 36.5 ± 1.5 °C, with that temperature being controlled by a thermometer connected to a resistor. Prior to the test, the specimens were exposed to the environment for 48h before starting to apply the mechanical loading.

Cracked specimens were firstly precracked by fatigue, while notched specimens were machined with four different notch radii of 0.25mm, 0.50mm, 1.00mm and 2.00mm for this work. To achieve greater accuracy and reproducibility in the tests, two of them have been performed per condition.

4. Results

The results of the tests are gathered in Table 3. From these K^N_{IEAC} results, according to LM, it is possible to predict the values of the apparent crack propagation threshold in an aggressive environment for notched components for a given notch radius. For this, K_{IEAC} was considered as the average of the two values from tests on cracked specimens (i.e., K_{IEAC}=13.85 MPa·m$^{1/2}$), and the least squares methodology was used to derive L_{EAC} (L_{EAC} being the fitting parameter).

According to the least squares fitting, L_{EAC}=0.465 mm. Thus, using equation (5), LM provides K^N_{IEAC} predictions represented together with the experimental results in Figure 6.

As can be observed, the experimental results showed an increase in the K^N_{IEAC} as the notch radius increased. This behaviour is correctly defined by the predictions that LM provides. The increase in the crack propagation threshold of precracked specimens (K_{IEAC}=13.85 MPa·m$^{1/2}$) compared to the one from a notched specimen of 2.00 mm of radius (K^N_{IEAC}=18.65 MPa·m$^{1/2}$) was close to 35 % when considering average values.

Table 3. Experimental results.

ρ (mm)	P_Q (KN)	K^N_{IEAC} (MPa·m$^{1/2}$)
Crack (0.00)	7.33	13.73
	8.33	13.96
0.25	9.12	15.75
	9.41	16.25
0.50	9.53	16.46
	10.38	17.94
1.00	10.56	18.24
	10.60	18.31
2.00	9.86	18.12
	10.44	19.18

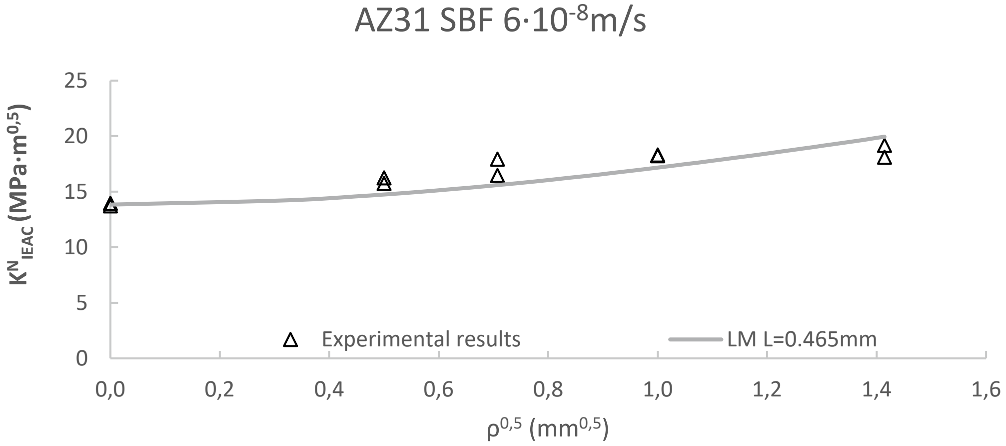


Fig. 6. Experimental results and predictions of K^N_{IEAC} provided by the LM.

5. Conclusions

In this work, the AZ31 Mg alloy has been studied for use as biomaterials in human implants. Its behaviour has been analysed in an aggressive environment that simulates human fluids and in the presence of notches of different radii. The Theory of Critical Distances has been used in the form of the Line Method to predict the behaviour of the material against the Environmental Assisted Cracking typical from the combination of stresses, aggressive environment and susceptible material.

AZ31 alloy experienced a clear notch effect, manifested as an increase in the value of the apparent crack propagation threshold (K^N_{IEAC}) as the notch radius grows. This increase is close to 35% when comparing the smallest notch radii (crack) and the largest radii studied (ρ = 2.00 mm).

The Environmental Assisted Cracking proposed methodology analysis based on the Theory of Critical Distances, specifically on the Line Method for this work, has been used together with the value of the critical distance that provided the best least squares fit, L_{EAC}=0.465mm, accurately reproducing the behaviour of the material in the analysed conditions.

References

Akahori, T., Niinomi, M., Fukunaga, K.I., Inagaki, I., 2000. Effects of microstructure on the short fatigue crack initiation and propagation characteristics of biomedical α/β titanium alloys. Metallurgical and Materials Transactions A 31 1949–1958.

Albrektsson, T., Brånemark, P.I., Hansson, H.A., Lindström, J., 1981. Osseointegrated titanium implants: Requirements for ensuring a long-lasting, direct bone-to-implant anchorage in man. Acta Orthopaedica 52, 155–170.

Chen, Q., Thouas, G.A., 2015. Metallic implant biomaterials. Materials Science and Engineering: R: Reports 87, 1–57.

Cicero, S., Madrazo, V., Carrascal, I.A., 2012. Analysis of notch effect in PMMA using the Theory of Critical Distances. Engineering Fracture Mechanics 86, 56–72.

Creager, M., Paris, P., 1967. Elastic field equations for blunt cracks with reference to stress corrosion cracking. International Journal of Fracture Mechanics 3, 247–252.

Engh, C.A., Bobyn, J.D., 1988. The influence of stem size and extent of porous coating on femoral bone resorption after primary cementless hip arthroplasty. Clinical Orthopaedics and Related Research 231, 7–28.

Ginebra, M.P., Traykova, T., Planell, J.A., 2006. Calcium phosphate cements as bone drug delivery systems: A review. Journal of Controlled Release 113, 102–110.

González, P., Cicero, S., Arroyo, B., Álvarez, J.A., 2019a. A Theory of Critical Distances based methodology for the analysis of environmentally assisted cracking in steels. Engineering Fracture Mechanics 214, 134–148.

González, P., Cicero, S., Arroyo, B., Álvarez, J.A., 2019b. Environmentally assisted cracking behavior of S420 and X80 steels containing U-notches at two different cathodic polarization levels: An approach from the theory of critical distances. Metals 9, 570.

Hanawa, T., 2010. Overview of Metals and Applications, in: "*Metals for Biomedical Devices*". Elsevier, pp. 3–24.

Hänzi, A.C., Sologubenko, A.S., Uggowitzer, P.J., 2009. Design strategy for new biodegradable Mg-Y-Zn alloys for medical applications. International Journal of Materials Research 100, 1127–1136.

ISO 7539: Corrosion of metals and alloys, Stress corrosion testing, Parts 1 to 11. International Organization for Standardization, Geneva, Switzerland, 2015.

Jacobs, J.J., Gilbert, J.L., Urban, R.M., 1998. Corrosion of metal orthopaedic implants. The Journal of Bone and Joint Surgery. American 80, 268–282.

Jafari, S., Harandi, S.E., Singh Raman, R.K., 2015. A review of stress-corrosion cracking and corrosion fatigue of magnesium alloys for biodegradable implant applications. JOM: the journal of the Minerals, Metals & Materials Society 67, 1143–1153.

Jafari, S., Raman, R.K.S., Davies, C.H.J., Hofstetter, J., Uggowitzer, P.J., Löffler, J.F., 2017. Stress corrosion cracking and corrosion fatigue characterisation of MgZn1Ca0.3 (ZX10) in a simulated physiological environment. Journal of the Mechanical Behavior of Biomedical Materials 65 634–643.

Jafari, S., Raman, R.K.S., Davies, C.H.J., 2018. Stress corrosion cracking of an extruded magnesium alloy (ZK21) in a simulated body fluid. Engineering Fracture Mechanics 201, 47–55.

Kokubo, T., Takadama, H., 2006. How useful is SBF in predicting in vivo bone bioactivity?. Biomaterials 27, 2907–2915.

Li, N., Zheng, Y., 2013. Novel Magnesium Alloys Developed for Biomedical Application: A Review, Journal of Materials Science and Technology 29, 489–502.

Long, M., Rack, H.J., 1998. Titanium alloys in total joint replacement - A materials science perspective. Biomaterials 19, 1621–1639.

Long, P.H., 2008. Medical Devices in Orthopedic Applications. Toxicologic Pathology 36, 85–91.

Mohajernia, S., Pour-Ali, S., Hejazi, S., Saremi, M., Kiani-Rashid, A.R., 2018. Hydroxyapatite coating containing multi-walled carbon nanotubes on AZ31 magnesium: Mechanical-electrochemical degradation in a physiological environment. Ceramics International 44, 8297–8305.

Neuber, H., 1958. Theory of notch stresses: principles for exact calculation of strength with reference to structural form and material, Springer. Verlag, Berlin, Germany

Peron, M., Torgersen, J., Berto, F., 2017. Mg and its alloys for biomedical applications: Exploring corrosion and its interplay with mechanical failure. Metals 7, 252.

Peron, M., Torgersen, J., Berto, F., 2019. Effect of Zirconia ALD coating on stress corrosion cracking of AZ31 alloy in simulated body fluid, Procedia Structural Integrity 18, 538–548.

Peterson, R.E., 1959. Notch sensitivity, in "*Metal Fatigue".* McGraw Hill, New York, USA, pp. 293–306.

Susmel, L., Taylor, D., 2008. On the use of the Theory of Critical Distances to predict static failures in ductile metallic materials containing different geometrical features. Engineering Fracture Mechanics 75, 4410–4421.

Susmel, L., Taylor, D., 2010. An Elasto-Plastic Reformulation of the Theory of Critical Distances to Estimate Lifetime of Notched Components Failing in the Low/Medium-Cycle Fatigue Regime, Journal of Engineering Materials and Technology 132, 021002.

Taylor, D., 2007. The Theory of Critical Distances: A new perspective in fracture mechanics. Elsevier, Oxford, UK.

Teoh, S.H., 2000. Fatigue of biomaterials: A review. International Journal of Fatigue 22, 825–837.