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MECHANICAL DESIGN FOR HIGH-PRESSURE REACTOR TO MICROWAVE-ASSISTED LEACHING OF REFRACTORY METALS

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ABSTRACT

The Colombian platinic alluvium metal contains platinum, iridium, osmium and palladium. Iridium and osmium confer a refractory condition that makes the leaching of the alluvium very difficult. This is the first aspect to be solved in the development of a refining process. Acid microwave-assisted leaching offers a higher leaching rate with high recovery levels, so it seems a possible way to solve this problem. A low-cost reactor to microwave-assisted leaching process has been designed using section VIII ASME code, comparison with commercial reactors and both SolidWorks and Ces-Edupack CAE. The 180 ml reactor was design take in account a high pressure of 8.3 MPa (1200 psi), 180°C temperature, chemical resistance to aqua regia, hydrochloric and hydro nitric acid and ergonomic and safety demands.

Keywords: high pressure reactor design, leaching microwave.

INTRODUCTION

The platinum group PMGs, consists on Pt, Pd, Rh, Ir, Ru and Os [1] and are considered both noble and refractory metals, meaning they are resistant to chemical attack in strongly oxidizing environments; for this, they are frequently used in medicine [2] and chemical and oil industries [3]-[5]. Also, they exhibit catalysts properties exploited by the automobile industry [6]. Their high melting point and high electrical conductivity give them a special use in electricity and electronic application. In Colombia, the PMGs is found as alluvium metal but process refining is not carried out due to lack of the required technologies and, also the alluvium nuggets contains Os and Ir, which confers a refractory characteristic that cause a great resistance to strong oxidizing acids.

Therefore, mining regional companies sell these as raw metals. Technologies for refining these metals include complex hydrometallurgical processes such as dissolution, precipitation, hydrolysis, distillation, organic precipitation, solvent extraction or ion exchange, molecular recognition technology and metal reduction, among others [7]-[11]. Regarding the first stage, that of dissolution; it been determined that microwave-assisted leaching is the fastest method, and therefore the most energy-efficient way to dissolve these elements, it even obtained a 100% dissolution in a single stage and in quite short timespans [12]. Said dissolution is generally made in highly oxidizing media such as hydrochloric acid, nitric acid or aqua regia in a container that in addition to being inert to these reagents must resist extremely high pressures generated by the oxidizing agents. Some developments of special reactors that resist these conditions are shown in Table-1.

International standards for high pressure vessels design generally take in account metals to its building. However, metals are not transparent to microwaves. Moreover, the reactors of the Table-1 are design to applications mostly related to foods or organic uses, and their use is not regulated for metal leaching. It exists a lack of standards to the design of this type of vessels. The present study intends to design a pressure vessel to provide a solution to the leaching stage of refractory platinic alluvium that is both economically and technically viable for mining companies take in account design international standards. Pressure vessels are sealed containers capable of holding a fluid (liquid or gaseous) at a temperature and pressure generally higher than that of the environment. To reduce the risk of failure in any of the aforementioned, different organizations have developed a series of documents such as codes, standards, and good practices used for their design and manufacture.

ASME Code - Section VIII

The ASME Code, Section VIII, considers containers for internal and/or external pressures, which can be generated by an external source or by the application of heat from a direct and/or indirect source. For this section, three partitions are made. Part 1 does not require a detailed evaluation of stresses as it calculates using a conservative safety factor of 4 when using tensile stress or 1.6 for yield strength. Maximum design pressure is 20.68 MPa (3000 psi). Part 2 performs a more specific evaluation, using higher allowable stresses, and using the maximum distortion energy theory (Von Mises). The design pressure is greater than 20.68 MPa and the safety factors are 3 for the tensile strength or 1.5 for the yield strength. Part 3 applies a very high pressure on the reactors, around 68.95 MPa. [13] [14] [15] [16].

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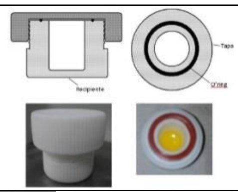
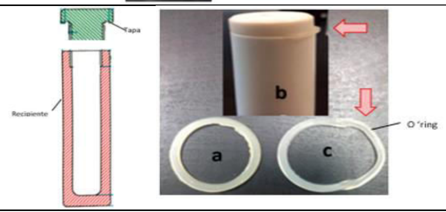
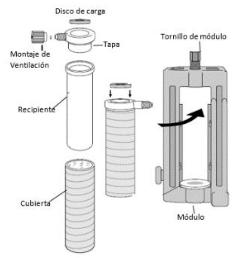
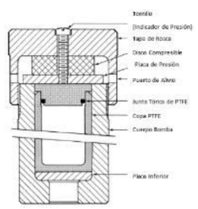
Table-1. Reactors to microwave-assisted leaching.

Characteristic	Design
Material: Polytetrafluorethylen (PTFE) Volume: 2 ml Transparent to microwave Resistant to strong oxidizing reagents Thread sealing with gasket Sample: Micro [17].	
Thick wall with a polymer gasket Material: Silicone as safety valve Use: environmental and food [18]	
Maximum Pressure: 5.5 MPa - 800 psi Maximum Temperature: 240°C Volume: 100 ml Weight of sample: 0.5 grams Material: Teflon® TFM Producer: CEM Teflon® TFM vessels XP-1500 plus high pressure Teflon® TFM vessels Use: Organic samples [19]	
Maximum Pressure: 8.3 MPa - 1200 psi Maximum Temperature: 250°C Producer: Parr Instrument Company [20].	

BS 5500 Unfired Fusion Welded Pressure Vessel

The "British Standards Institution" document is applied to pressure vessels not subjected to flame action or fusion welded. It contains the design, manufacture and inspection. The safety factor is 2.35 using the yield strength. [21] [22] [23].

AD2000 - Merblatter

The German code expresses its equations for shell thicknesses under welded internal pressures. The suggested safety factor is 1.5 in relation to the yield strength. This German standard defines the design and construction of vessels at a pressure less than 30 MPa (4351 psi) and uses a safety factor of 1.66 in relation to yield strength. [21] [24]

JIS B 8265

This Japanese standard defines design and construction of vessels at pressure less than 30 MPa (4351 psi) and uses a safety factor of 1.66 in relation to yield strength [25] [22].

Table-2 shows some of those documents and the organizations that use them as guidelines. Table-3 shows a comparison of the different codes with their safety factors and maximum design pressures.

Table-2. International standards for high pressure vessels design (Arredondo n.d.).

Land	Code or document	Institution
U.S	ASME - Secc. VII Boiler & Pressure Vessel Code	ASME
United Kindom	BS 5500 Unfired Fusion Welded Pressure Vessel	BSI
Germany	AD2000 – Merblatter	AD
Italy	ANCC	ANPCPC
Netherlands	RTOD / Regeis Voor Toestellen	DS
Switzerland	Tryekkarlskommissionen	SPVS
Australia	AS 1210: Unfired Pressure Vessel	SAA
Belgium	IBN Construction Code for Pressure Vessel	BSI
Japon	JIS B 8265	MITI
France	CODAP/ SNCT Construction Code for Unfired Pressure	SNCTI
China	GB 150	SAC
European Union	EN 13445 – Directiva 97/23/CE	PE
India	IS 2825	BIS
Korea	Kepic MG	KEA
Russia	Gost R 52857	EASC
New Zealand	NZ 1210	NZS
Canada	B5.1-R1	CSA

Table-3. Safety factor, mechanical properties used and pressure according to the international standard.

Code or document	Safety factor	Pressure
ASME VIII, Div. 1	4 – Tensil strength or 1.6 - yield strength	Maximum 20.68 MPa
ASME VIII, Div. 2	3 – Tensil strength or 1.5 - yield strength	Maximum 20.68 MPa
ASME VIII, Div. 3	-----	Maximum 68.95 MPa
BS 5500	2.35 - yield strength	----
AD2000 – Merblatter	1.5 - yield strength	----
JIS B 8265	1.66 - yield strength	Maximum 30 MPa

MATERIALS AND METHODS

The design of the leaching reactor was carried out by determining the design, functional, and ergonomic parameters. Choosing the best method for calculating the dimensions according to the standards ASME-Division I, BS 5500 Unfired Fusion Welded Pressure Vessel, AD2000 - Merblatter, JIS B 8265 and selecting materials using the Ces-EduPack software. Then a mechanical simulation was done in Solidworks.

RESULTS AND DISCUSSIONS

Design settings, calculations and simulation parameters are defined to determine and validate the design as follow

Design Parameters

Within the design parameters, two types of settings are defined, those related to mechanical and thermal properties, and those related to reactor handling and safety (see Figure-1).

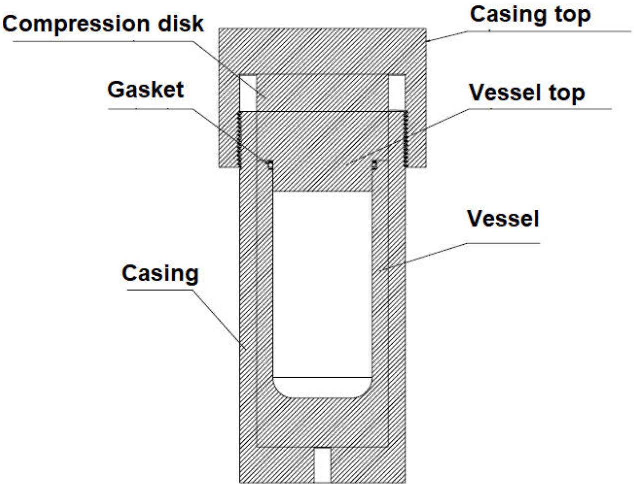


Figure-1. Reactor design.

Mechanical and Thermal Design Parameters

- Microwave transparent material resistant to aqua regia [12]. Aqua regia is a combination of nitric and hydrochloric acid, the most efficient ratio is 3: 1 HCl: HNO3 [26].
- Leaching temperature is between 150° C and 210° C [12]. Microwave leaching reactors on the market have maximum working temperatures up to 250° C. [20][19].
- Maximum working pressure; this will depend on the vapor pressure of the aqua regia at the leaching temperature. Some commercial reactors have maximum working pressures of 2.4 MPa (350 psi), 5.5 MPa (800 psi) and 8.3 MPa (1200 psi). For this design, the highest design pressure was chosen as 8.3 MPa [20][19].

Ergonomic and Safety Parameters

- Safe and easy to open/close reactor top.
- Static confinement design packaging, which allows a hermetic sealed of the chamber and top.
- Casing and casing top, designed to provide the mechanical properties necessary to withstand the design pressure. A fine pitch metric thread is designed into the casing for seal.
- Liner cap compression disc, this together with the cap threaded to the liner, will allow the shutting between the reactor and its top.

Material Selection

The selection of the material was carried out using the Ashby methodology and CES EduPack software. The properties mentioned above was considering to the selection. Only polymeric materials were selected, because metals reflect the wavelength of microwave radiation and ceramics show brittleness, and difficult or no machinability [27], [28]. Four possible materials were obtained for the body, the top and the seal gasket of the reactor. Nine was obtained for the casing. When conducting a market study in Colombia, the selected materials was:

- Polytetrafluoroethylene (PTFE) → reactor, reactor top, gasket and disk. Yield strength 19.7 MPa (2857.24 psi) [29]. Maximum service temperature: 260 ° C [30].
- Polyoxymethylen, acetal (homopolymer) (POM-H) → casing, casing top, Yield strength 65.5 MPa (9500 psi) [29]. Maximum service temperature: Short Cycle 150°C, Large Cycle 90°C [31]

Reactor Design

Reactor Chamber

From Table-2 the 8.3 MPa (1200 psi) design pressure, it was defined to use the recommendations of the ASME VIII, Div. 1 code [32] to perform the reactor design calculations. This code suggests steel as manufacturing materials, but for this specific case, metallic materials are not applicable due to their lack of efficiency in transmitting microwaves. The design pressure calculation is made up of the fluid’s static pressure and the maximum working pressure. The fluid’s static pressure [33] is calculated according to equation 1 and the maximum working pressure that which the fluid will reach during the process.

$$P_{static} = HHLL * g * \rho_{H_2O} * SG_{fluid} \quad (1)$$

Where
Pstatic : Static pressure
HHLL : High level liquid; 0.08 m
g : gravity; 9.81 m/s2
ρH2O : water density 997 kg/m3
Sgfluid : specific gravity of fluid, 1.21
Then, Pstatic = 0.0009 MPa (0.13 psi)

The design pressure will be the sum of the fluid’s static pressure plus the maximum working pressure reached in the process. However, considering that the maximum operating pressure is 8.3 MPa (1200 psi), 0.0009 MPa (0.13 psi) is negligible. To find the design pressure, equation 2 [34] is applied, which includes a safety factor.

$$P = maximum(1{,}1P_0;\ P_0 + 2) \quad (2)$$

Where:
P : Design pressure
P0 : Maximum service pressure 8.3 MPa (1200 psi)

P = maximum (9.13 MPa; 8.28 MPa)
Then, P = 9.13 MPa (1320 psi)

The calculation of the maximum allowable strength is given by equation 3 [35], [36].

$$\sigma_{asd} = \frac{\sigma_{ys}}{S.F} \quad (3)$$

Where:
σasd: Allowable strength design
σys: Yield Strength for PTFE is 19.7 MPa (2857.24 psi) and for POM-H is 65.5 MPa (9500 psi)
S.F: Safety factor 1.6 as ASTM VIII Div.1
Then
σasd = 12.31 MPa (1785.8 psi) for PTFE
σasd = 40.94 MPa (5937.49 psi) for POM-H

For the calculation of the reactor's wall thickness, the ASME VIII Div. 1 code was used in its section UG-27 where some equations for the thickness of casings under internal pressures in pressure reactors are suggested. These are selected by comparing them with the design pressure (P), as shown in Table-4.

As evidenced, the longitudinal stress is the one that satisfies the design condition, and leads to equation 4:

$$t_{wall} = \frac{PR}{2SE - 0.4P} \quad (4)$$

Where
P : Design pressure; 9.13 MPa (1320 psi)
R : Internal ratio; 24 mm (0.94 in) for 180 ml volumen and 100 mm high
S : allowable strength; 12.31 MPa (1785.78 psi)
E : joint efficiency, 1 seamless
Twall : Wall thickness
Then, twall = 7.7 mm (0.3 in)

The thickness of the reactor bottom is calculated using the flat circular head equation 5, from section UG-34 of the ASME VIII Div 1 [13], [37].

$$t = d\sqrt{\frac{CP}{SE}} \quad (5)$$

Where
d : Internal ratio; 48 mm (1.88 in)
P : Design pressure; 9.13 MPa (1320 psi)
C : dimensionless factor, depends on the head fixation method, on this case; 0.33
S : allowable strength; 12.31 MPa (1785.78 psi).
E : joint efficiency, 1 seamless
T : Bottom thickness
Then, t =23.7 mm (0.93 in)

Table-4. Design condition for stress type.

Stress type	Design condition	Results	
		PTFE	POM-H
Circumferential	P< 0.385SE	4.74 MPa or 687.52 psi	15.76 MPa or 2285.9 psi
Longitudinal	P < 1.25SE	15.39 MPa or 2232.2 psi	51.17 MPa or 7421.8 psi

*Design pressure 9.13 MPa (1320 psi), S allowable strength, E joint efficiency

To define the internal rounding that can be seen in Figure-2, the following code conditions are used [13]:

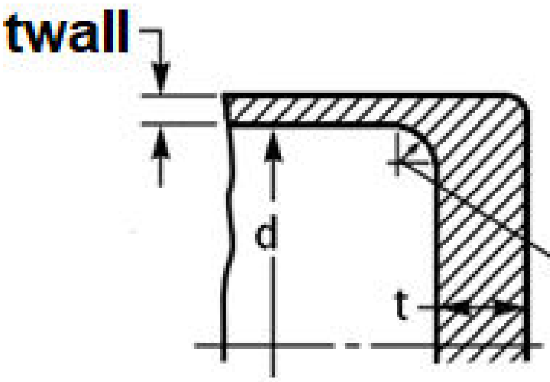


Figure-2. Rounding between flat circular head and wall.

r = 10 mm (0.375 in) for twall ≤ 38 mm (1½ in)
r = 0.25 twall for 38 mm (1½ in) > twall > 19 mm (¾ in)
Then, rounding on the inside is 10 mm (0.375 in) according to twall ≤ 38 mm (1½ in)

Reactor Top, Casing and Top Casing

The design of the other elements such as the reactor top, the causing body and the top causing were carried out following the ASME VIII Div. 1 code, with the steps and equations used previously in the calculation of the reactor body. The reactor top was designed as a plug due to the casing form and to prevent leakage on the gasket. The same bottom thickness of 23.7 mm (0.93 in) was used with an outer radius of 31.8 mm (1.25 in) and a run-in length of 15 mm (0.59 in). For the casing and its top, the following initial conditions were defined (Table-5).

Table-5. Initial condition for stress type.

Casing	Internal radio	High	Material
Body	31.8 mm (1.25 in)	179.6 (7.07 in)	POM-H
Top	40 mm (1.57 in)	66.1 mm (2.6 in)	POM-H

To determine the thickness of wall and top of casing, the two conditions mentioned in Table-4 were met, being the circumferential condition the greater, its equation is shown:

$$t_{wall} = \frac{PR}{SE - 0.6P} \quad (6)$$

In Table-6 the results can be observed.

Table-6. Thickness wall and bottom of casing.

Casing	Wall thickness	Bottom thickness
Body	8.1 mm (0.32 in)	17.2 mm (0.67 in)
Top	10.7 mm (0.42 in)	22.1 mm (0.87 in)

Gasket

The gasket will be made using PTFE, since it will be exposed to vapours or fluids such as aqua regia. Gasket of this material are not in stock; therefore, its mechanization is necessary [29]. Its design is performed in conjunction with the housing design using two dimensions: W section and external diameter DE [38], [39] as shown in Figure-3 (a). The housing is designed in the reactor body and the depth L, the width G and the radius R are considered as shown in Figure-3 (b).

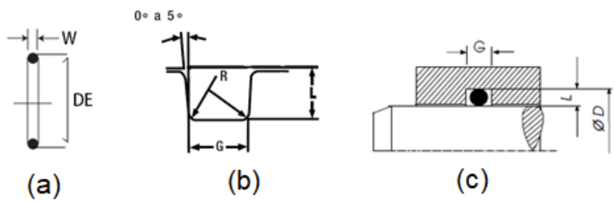


Figure-3. (a) Gasket dimmensions [40], (b) Outer diameter of gasket (Parker 2019), (c) Diameter of packing (Parker 2019).

The dimensions of the housing must guarantee a crushing force for static use that varies from 12% to 25% and dynamic use of deformation from 8% to 20%. The dimension "L" must guarantee a deformation of the section "W" not less than 0.25 mm in absolute value and is a function of the internal diameter of the reactor [38], the gaskets have a section W of 2.65 mm defined according to the internal diameter and using the criteria given by the producers of Global O-Ring and Seal seals [39].

Using Table-7, the measurements depth L, width G and rounding R can be obtained, with L = 2.15 mm, G = 3.7 mm and R = 0.4 mm. The conventionally defined angle is 5°.

Table-7. Thickness wall and bottom of casing.

Secc.W	Hausing dimensions			
	Depth L		Guide G ±0.1	Radio R
	Static	Dinamic		
1.78	1.25 1.35	1.40 1.45	.0.5	0.1 0.4
2.62	2.05 2.15	2.25 2.30	30.7	0.1 0.4
3.53	2.80 2.95	3.05 3.10	40.9	0.2 0.6
5.33	4.30 4.50	4.65 4.75	70.3	0.5 1.0
6.99	5.75 5.95	6.00 6.10	90.7	0.5 1.0

To determine the diameter of packing D (Figure-3 (c)), the internal diameter of the reactor (48 mm) is added with twice the measurement of L, resulting in 52.53 mm.

Thread, Screw and Nut

The force acting on the fine pitch metric thread is determined, using the following equation.

$$P = \frac{F}{A} \quad (7)$$

Where

A : It is the area of the compression disc that will be in contact with the top of the casing. 3166.92 mm2 (4.9 in2)

F : clear force of the equation. [35]

Then, F=28829.91 N (6481.2 lb)

The design is determined according to equations 8, 9 and 10 and as shown in figure 6 [41], [42].

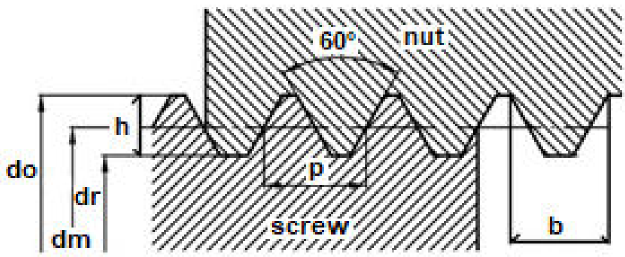


Figure-4. Fine pitch metric thread (National Technological University).

Equations 8 is used to find the mean diameter,

$$dm = do - (0.649519 * p) \quad (8)$$

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Where
do = 81 mm (3.18 in)
p = 2 mm (0.078 in)
Then, dm = 79.7 mm (3.137 in)
Equation 9 allows finding the root diameter,

$$dr = do - (1.082532 * p) \tag{9}$$

Then, dr =7 8.8 mm (3.10 in) and equation 10 allows knowing the height of the tooth

$$h = 0.613435 * p \tag{10}$$

Then, h=1.227 mm (0.048 in)

Thread Height

Table-8 shows the equations [43] 11 to 15 for the height of the thread according to the forces present in the thread of the screw and nut.

Table-8. Thread height.

Stress on the component		Equations	Results
Contact pressure	Screw and nut	$H = \frac{F * p * N}{\pi * dm * h * Syc}$ (11)	8.8 mm (0.34 in)
Bending stresses in the fillet	Screw	$H = \frac{3 * F * h * p * N}{\pi * dr * b^2 * Syt}$ (12)	18.9 mm (0.74 in)
	Nut	$H = \frac{3 * F * h * p * N}{\pi * do * Syt * b^2}$ (13)	18.4 mm (0.72 in)
Shear stress at the root of the fillet	Screw	$H = \frac{3 * F * p * N}{2\pi * dr * b * Sys}$ (14)	16.2 mm (0.63 in)
	Nut	$H = \frac{3 * F * p * N}{2\pi * do * b * Sys}$ (15)	26.3 mm (1.03 in)

Based on the results obtained, we select the highest result being 26.3 mm (1.03 in) in height of the thread. This occurs at the root of the fillet in the nut. Additionally, a disk that deforms elastically and ensures the hermetic closure of the reactor top during the process was designed. The 31.75 mm (1.25 in) diameter and 18.03 mm (0.71 in) height, made of PTFE.

Mechanical Simulation

Static mechanical analysis was performance in SolidWorks software with the initial conditions mentioned in Table-9. The meshed can be appreciated in Figure-5 (a).

Table-9. Simulation parameter.

Mesh Type	Solid Mesh
Mesh used	Standard mesh
Element size	5.13342 mm
Tolerance	0.256671 mm
Mesh quality plot	High-order quadratic elements
Total number of nodes	84660
Total number of elements	57450
Maximum aspect ratio	9.0893

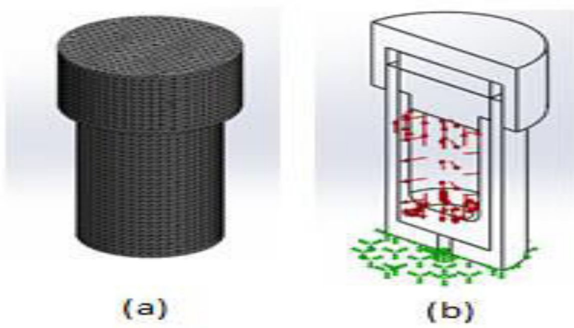
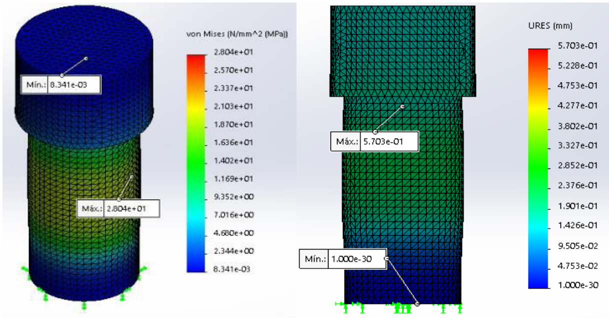


Figure-5. Mesh and load on the reactor.

In this simulation, Von Mises stress was analyzed, safety factor that it has respecting the construction material is calculated, and the maximum displacements due to pressure were also analyzed. The simulation was made from the reactor's assembly, a pressure load of 9.13 MPa (1320 psi) was used inside the reactor as seen in Figure-5 (b) and a fastening at the base of the reactor. The maximum and minimum Von Mises stress obtained by the simulation were 28.94 MPa (4197.39 psi) and 8.341×10^{-3} MPa (1209.75 psi) respectively. As can be seen, the minimum stress is negligible. Comparing with the yield point of the POM-H material (65.5 MPa - 9499.97 psi) it is evidenced that there is a safety factor of 2.26, indicating that it will withstand the design pressure twice. Figure-6 (a) shows that reactor has a small zone of the maximum Von Mises value according to the color scale, appearing on the body of the casing. Figure-6 (b) shows the location of the resulting displacements in the reactor. According to the color scale, the displacement zones are very small, since most of the

ARPN Journal of Engineering and Applied Sciences

body is in blue and green, with minimal displacement. The maximal displacement was calculated as 0.57 mm and the minimum was $1x10^{-30} \cong 0$ mm.



(a) (b)

Figure-6. (a) Von Mises stress (b) displacements in the reactor.

CONCLUSIONS

The material selected for the body, cover and packaging of the reactor was PTFE because it is transparent to microwaves, resistant to strongly oxidizing environments, and it has defined mechanical stress requirements, for the jacket it was POM-H since it is not exposed to acids and must ensure greater mechanical resistance.

A reactor body and jacket were designed for a working pressure of 9.13 MPa (1320 psi), with a total volume of 180 ml and a maximum temperature of 250° C, following the design conditions given by the ASTM VIII Division standard 1.

As it is a guided design under division 1 of the ASME code, it is a conservative design when considering greater thicknesses, and greater design pressure, since it includes a safety factor for its design, as well as the material. However, compared to other pressure vessel design codes, the ASME code has low safety factors.

The simulation gives us an elastic deformation as a result, since the value of the Von Mises stress is less than the value of the yield stress of the material.

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