

THERMAL AND STRUCTURAL ANALYSIS FOR A REACTOR USED IN HYDROTHERMAL SYNTHESIS UNDER SUPERCRITICAL CONDITION

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ABSTRACT: This paper presents a thermal and structural analysis for hydrothermal reactor under high pressure and high temperature operating condition. The study was performed using ANSYS software to obtain the stress and strain for equipment used in hydrothermal synthesis in crystal growth process. This study was performed for different temperatures in variation domain from 300°C to 500°C and different pressure from 100 MPa to 250 MPa. The stress results obtained from numerical simulation show the maximum loadings and equipment behaviour in high pressures and temperatures condition. The thermal analysis obtained show the heat flux in reactor body and also this thermal analysis was coupled with structural analysis to obtain the very good agreement with the normal operating condition from hydrothermal synthesis. In conclusions, this paper presents a numerical analysis regarding the reactor behaviour in hydrothermal process.
KEY WORDS: Hydrothermal autoclave, High-pressure, ANSYS

1. INTRODUCTION

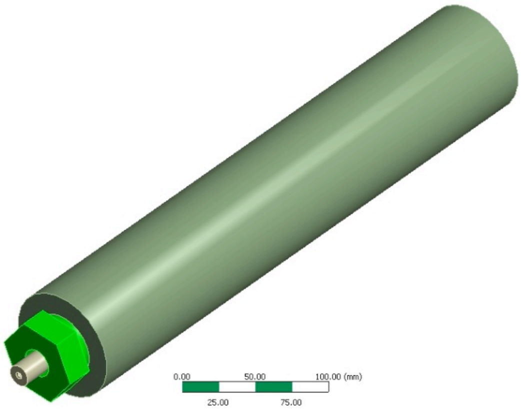
Piezoelectric single crystals are used in frequency control and acoustic wave devices. The quality of the crystal determines the performance of these devices [1,2]. Hydrothermal growth is the method of preference to grow high quality single crystals. Most of the piezoelectric single crystals are obtained using the hydrothermal growth technique, which takes place in an autoclave at high temperatures (around 400 °C) and high pressure (1000 bar) [1]. Using critical water conditions, namely temperature and pressure of 374 °C and 22.1 MPa, respectively, the solvent properties for many compounds, such as dielectric constant and solubility, change dramatically under supercritical conditions [3]. There are a lot of materials obtained at the high temperatures and pressures such be: TiO_2 [4], SiO_2 dopet with Sn [5], $CoFe_2O_4$ [6], $BaTiO_3$ [7]. Thermoelectric materials with delafossit structure as $CuAlO_2$ [8] was obtained by hydrothermal method at high pressures and temperatures. Their advantage consists in the relatively lower reaction temperature (<400◦C) in comparison to general solid-state reaction (1100◦C, 24 h). When crystal growth occurs an autoclave is usually divided into two chambers by a baffle with an area opening ranging from 2% to 20% [9]. The lower chamber is maintained at a higher temperature at which the raw material is dissolved into the solution. The upper chamber is the growth chamber where the temperature is set lower and the crystal grows on seeds by precipitation from the supersaturated solution. The high pressure and high temperature environment in an autoclave can increase the solubility of quartz in aqueous solution and make crystals grow at practical rates. In our case, temperature is the same all over the autoclave because was used for obtain the thermoelectric materials $CuCrO_2$ with delafossitice structure. In this paper presents a numerical analysis of stress strain state of an autoclave used for hydrothermal synthesis. Numerical analysis was performed for the autoclave operating parameters namely pressure ranging from 100 MPa to 250 MPa and temperatures between 300-500 °C.

2. NUMERICAL SIMULATION

Numerical analysis was performed using ANSYS 12 finite element analysis program. 3D model of the autoclave was conducted in accordance with the dimensions and Solid Works program execution model real conditions shown in table 1 and imported into ANSYS for finite element analysis. 3D model is shown in figure 1. Figure 1 shows the physical model of the autoclave used for this experiment and this autoclave is also used in the numerical simulation.

Table 1. Sizes steel autoclave

Parameters	Size [mm]
Outer diameterD_O	25
Inner diameterD_I	80
HeightH_{total}	430



a)

b)

Figure 1. Industry hydrothermal autoclave: a) 3D CAD model autoclave, b) Working model autoclave

Number of finite elements used in the 3D mesh was 147,316 and the number of nodes was 219,405. Type the elements used for numerical analysis was elements of type 187 solid that contain the following features defined by 10 nodes having three degrees of freedom at each node: translations in the nodal x, y, and z directions. The element has plasticity, hyperelasticity, creep, stress stiffening, large deflection, and large strain capabilities [10]. We used elements with size 5 mm as a result of a study of convergence of the solution. Example of mesh model is shown in Figure 2.

The material it is made autoclave is a stainless steel X2NiCr25 21 according to DIN 17006 with the following characteristics presented in Table 2a, 2b [11]. Numerical analysis was performed by coupling thermal analysis with structural analysis. From thermal analysis performed at temperatures ranging from 300-500 °C was obtained heat flow generated in the autoclave body in different directions.

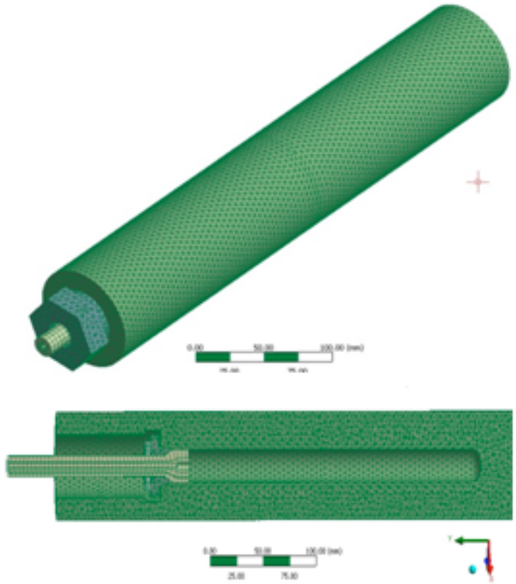


Figure 2. Discretized model of autoclave

Table 2. a. Component elements properties

Carbon [%]	0.25
Chromium [%]	24-26
Iron [%]	48-53
Manganese [%]	2
Nickel [%]	19-22
Phosphorous [%]	0.045
Silicon [%]	1.5
Sulfur [%]	0.030

Table 2. b. Properties of the materials

Density[g/cm^3]	8.00
Hardness, Brinell	170
Tensile Strength, Ultimate[Mpa]	655
Tensile Strength, Yield[MPa]	275
Elongation at Break[%]	45
Modulus of Elasticity[GPa]	200
Specific Heat Capacity[J/g-°C]	0.500
Thermal Conductivity[W/m-K]	14.2

Figure 3 shows the heat flow obtained if we have a pressure of 100 MPa and a temperature of 300 °C. Note that the heat flow is distributed approximately evenly across the entire autoclave.

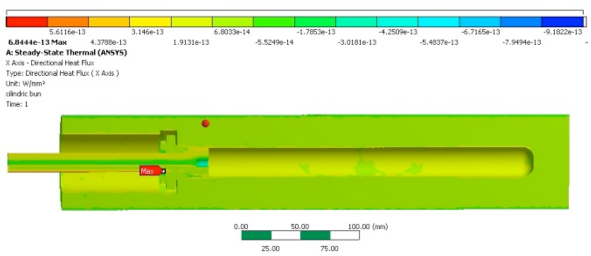
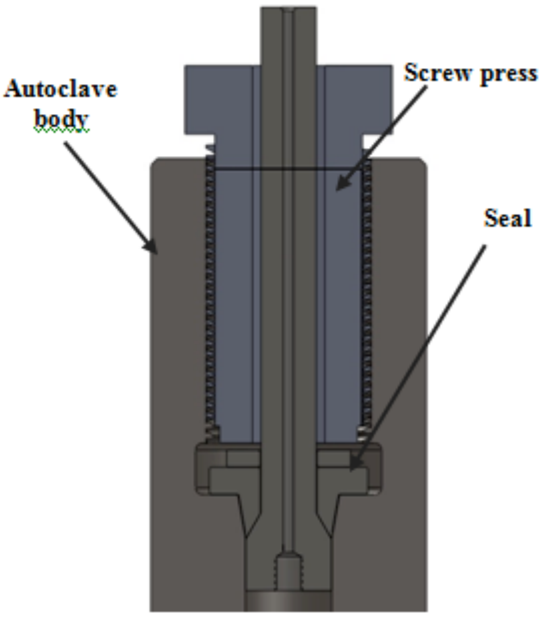
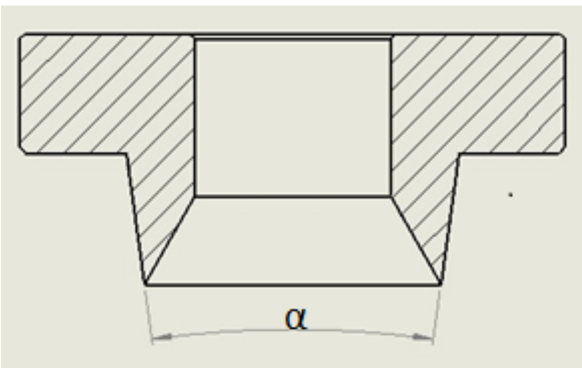


Figure 3. Directional Heat Flux in X direction at 300°C [W/mm^2]

For structural analysis has imported temperature for the entire autoclave thermal analysis, simulating actual operating condition. As boundary conditions imposed in the structural analysis has been imposed pressure inside the autoclave determined by measuring with pressure gauge attached to the end of the autoclave. As can be seen in figure 3D (figure 4) an important role in ensuring tightness is the seal.



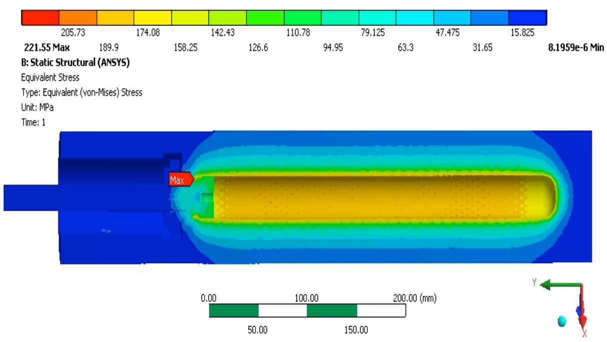
a)



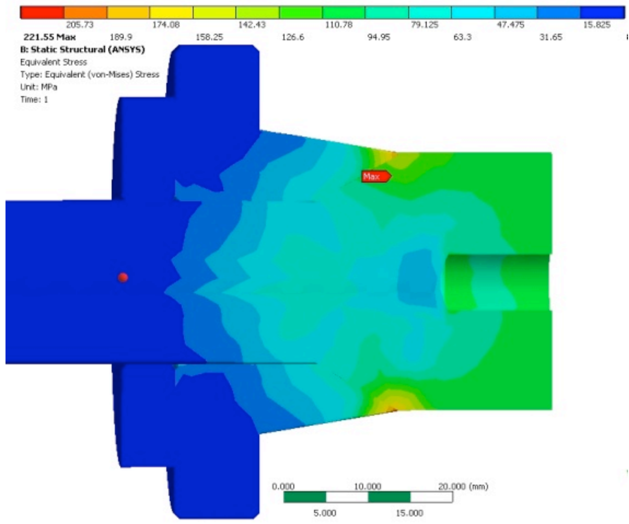
b)

Figure 4. Parts of the autoclave : a) Section in autoclave, b) Execution drawings of the seal

There have been termico-structural analysis for 2 seal configurations (α = 10°, 15°) shown in detail in Figure 4b to obtain the stress field respectively areas of high stress. In Figures 5, 6 and 7 presents the maximum equivalent stress, radial stress, radial and circumferential tension.



a)



b)

Figure 5. Maximum equivalent stress [MPa]: a) Autoclave body, b) Detail

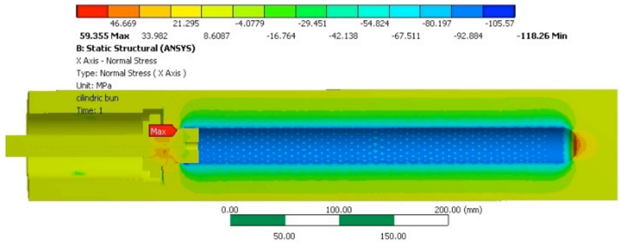


Figure 6. Radial stress [MPa]

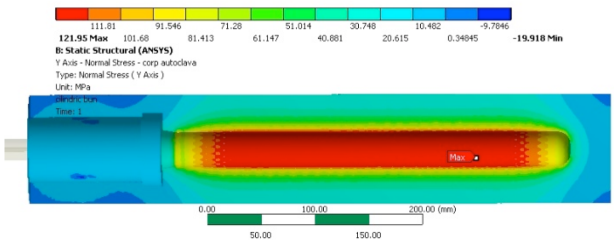


Figure 7. Circumferential stress [MPa]

When a 3D analysis, 3D tension is calculated based on the normal and shear exponent. Very often state of tension is expressed on the von Mises equivalent stress [12].

$$\sigma_v = \left[\frac{(\sigma_1-\sigma_2)^2+(\sigma_2-\sigma_3)^2+(\sigma_3-\sigma_1)^2}{2}\right]^{1/2}$$

In Figures 5, 6 it is observed that the maximum value of stress is reached in the contact area between the seal and piston. Maximum stress being equivalent to 221.55 MPa. Maximum value being 221.55 MPa for equivalent stress and 59,355 MPa for radial stress, close to the yield strength of 275 MPa at material. When radial stress can be observed as point up and the bottom autoclave. In case of circumferential stress in Figure 7 we can see that the maximum stress is reached inside the autoclave between contact from fluid and autoclave body. The maximum value of stress being 121.95 MPa. In numerical analysis, it was determined the value of the seal surface deformation shown in Figure 8 for working pressure of 100 MPa and a temperature of 300 ° C.

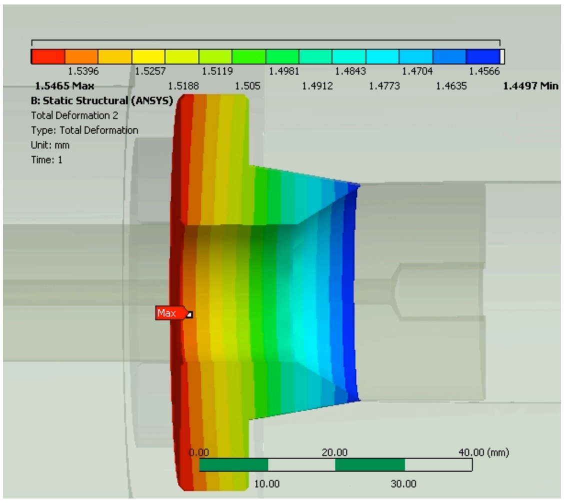


Figure 8. Total deformation on the seal to the angle α=10° [mm]

As shown in Figure 8 the maximum deformation occurs in the contact zone between the washer and seal surface so validated and in real working conditions. Reaching maximum strain value of 1.5 mm.

3. RESULTS AND CONCLUSIONS

The numerical analyzes performed for 400°C and 500°C with pressure ranging from 100 MPa to 250 MPa pressure was graphically represented pressures influence the temperatures analyzed, on maximum equivalent stress as shown in Figure 9. Is observed with increasing pressure at 400 °C, stess rises and it reaching the value of 700 MPa. In the case temperature of 500 °C and pressure of 1000 bar stress reaches a value of 275 MPa and with increasing pressure stress reaches almost the same values as in the temperature of 400°C.

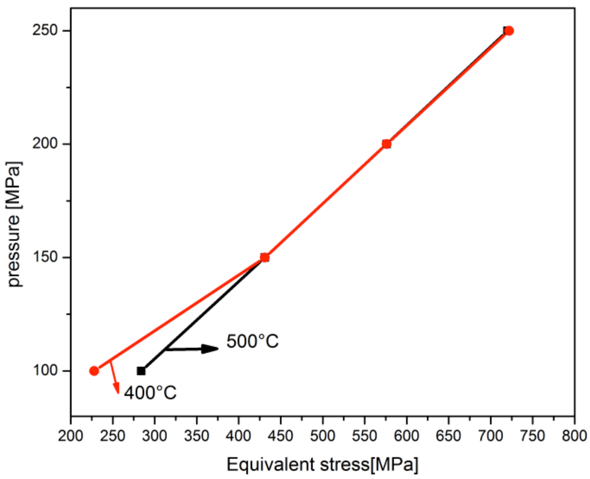


Figure 9. The results of equivalent stress depending on working pressure

From figure 10is observed in numerical analyses at a temperature 400°C, the radial stress increases with increasing working pressure, peaking to 275MPa.

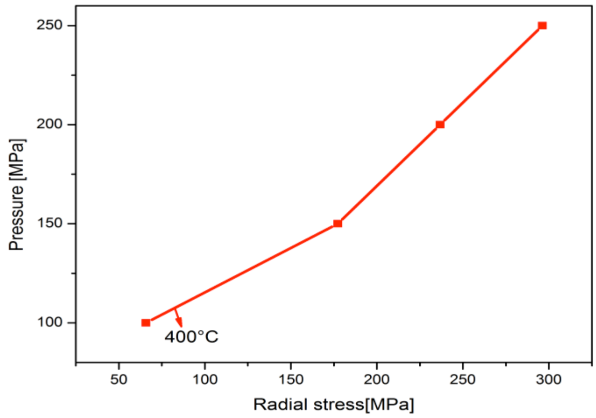


Figure 10. The results of radial stress depending on working pressure

Through changing the angle of the seal α=10°and α=15°, which performed tightness have been determined stress in the autoclave, the maximum stress obtained at contact fitting-body autoclave at a temperature of 300° C and pressures ranging between 100 and 250 MPa. Following results were represented curves of variation of equivalent stress according to the pressure and angle of the seal. It is seen from figure 11 that the stress increases depending on the angle sea land pressure working and in case the angle α= 15° pressure increases linearly. It can be said that the angle of inclination affects tightness system, influencing the maximum stress.

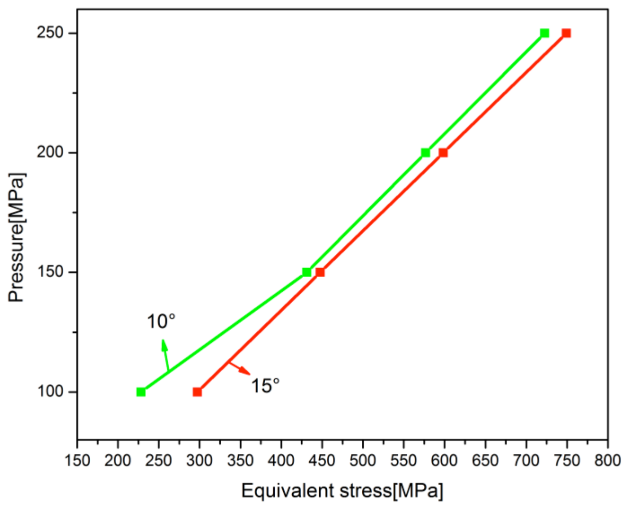


Figure 11. The results of radial stress depending on working pressure and inclination of the angle seal

Thermal flow resulting from the analysis of temperature to 300 ° and 400 ° C can say that is also influenced by the working pressure as shown in Figure 12.

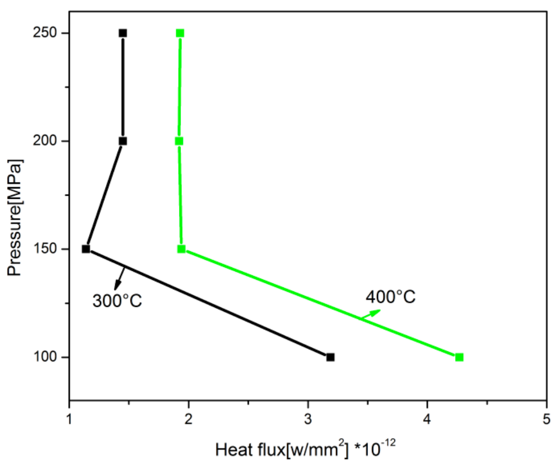


Figure 12. The results of heat flux depending on pressure and working temperature

4. CONCLUSIONS

This paper presents thermal-structural analysis of an autoclave with the working parameters pressure ranging between 100 and 250 MPa and temperatures between 300 and 500°C. From this work we can see the influence of pressure and temperature on the mechanical behavior of the autoclave. Maximum stresses occurring in the contact area between seal and autoclave body, being included in the material yield strength. When changing the the angle of inclination seal which performed sealing, is observed change in stress. Maximum deformations resulting from numerical simulations at different temperatures and pressures reveals that they are the gasket. This study provides the analysis of different configurations possible seal and requests that occur when operating. The review finds that if pressure exceeds 250 MPa are achieved high values of stress causing a possible collapse in autoclave operation, or failure due to loss of pressure tightness may occur.

5. ACKNOWLEDGEMENTS

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