CALCULATION OF BULK AND SHEAR MODULUS BY ANALYTICAL METHODS OF QUENCHED AISI 4140 STEEL AT DIFFERENT CONCENTRATIONS OF PVP

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Abstract. Quenching is a heat treatment that aims at raising the hardness and mechanical resistance of the materials. This process transforms the austenite into martensite by a microstructural change without diffusion. For this to run, it is necessary that the material is heated above the austenitization temperature and rapidly cooled in a fluid. Currently, fluids that blend water with polymers are being used for this heat treatment. The polymer concentration in solution is critical for changing the heat removal capacity and hence the formation of martensite. The concentration of polymers in the solution is fundamental for the alteration of the heat withdrawal capacity and consequently the formation of martensite. Often, there is partial formation of martensite and the material develops one or more secondary phases, giving rise to an inhomogeneous material, which causes the properties of the materials to change. The analytical curves allow the calculation of the properties of inhomogeneous materials, from previously known characteristics of the matrix materials and second phase. In this work, samples of AISI 4140 steel were treated in different concentrations of polymer solution based on PVP. From the identification and quantification of its phases, its bulk and shear modules were calculated by five different methods. These calculations resulted in specific curves for each method, which were compared to each other.

Keywords: Quenching, PVP, Analytical Curves, Bulk Modulus, Shear Modulus.

1. **INTRODUCTION**

The quenching process consists of heating a steel to its austenitization temperature, followed by rapid cooling in a fluid. The purpose of this heat treatment is to increase the mechanical strength and hardness of the material by the microstructural transformation of the steel, which must be martensitic at the end of the process. The transformation of austenite into martensite occurs by a non-carbon diffusion process, and for this to happen, the material needs to be cooled rapidly. In many cases, even if undesired, diffusion of the material occurs partially, resulting in more than one microstructural component (Totten, 2007).

The cooling of the material is carried out in fluids, usually liquids. When the heated material contacts the fluid, it develops three consecutive phases: steam blanket, boiling and convection. The steam blanket stage, the first one, is characterized by the formation of a vapor barrier around the part, which prevents the heat exchange by conduction and convection, resulting in low thermal changes and minimizing the possibility of transformation without diffusion (Hernandez and Morales, 2009). The process performed using traditional quenching fluids, such as water and mineral oil, breaks down this vapor layer always starts at the bottom of the part and is gradually raised to the top of the material. This progressive breakage of vapor film causes different thermal exchange rates throughout the body, resulting in different mechanical properties along the material (Koudil et. al., 2014). Furthermore, austenite transformation to martensite causes an increase in volume of the crystalline steel mesh, ranging between 2 and 4% (Magee and Davies, 1972). This expansion of the crystalline lattice causes residual stresses on the quenched parts, which are severely intensified by the gradual breaking of the vapor film, since there is nonuniform expansion along the body. These tensions when they are very high can trigger distortions and cracks in the tempered materials (Grum and Bozic, 2005).

Polymeric quench fluids dissolved in water are a good alternative for minimizing such damaging effects to tempered materials because their vapor film collapses instantly, failing to develop a gradual break. As a result, there is greater uniformity of thermal changes and minimization of residual stresses. Other positive effects of these fluids in relation to other solutions used for steel tempers are that they have low toxicity, biodegradability and are not flammable (Koudil et. Al., 2014). The main factor for changing the heat exchange capacity of these fluids is the concentration of polymer in the water. Increasing the polymer concentration in the blend causes a higher viscosity in the solution, making the vapor layer

thicker and hard to break, reducing cooling capacity. Thus, the higher the concentration, the lower the possibility of obtaining martensite, especially in the deeper layers of the body, since the interior of the body exchanges heat with the surface layers only by conduction, not directly contacting the cooling fluid (Ramesh and Prabhu, 2016).

Therefore, the core of the material cannot obtain a totally adifusional transformation, presenting one or more phases besides the martensite. These phases have different properties between them, which changes the final mechanical characteristics of the thermally treated material. Some final mechanical properties can be approximated by means of analytical equation (Hemming et al, 2003)

The approximations through the analytical equations are intended to predict the mechanical behavior of inhomogeneous materials, which have a matrix and another phase dispersed in it. The analytical equations of Mori-Tanaka, Voigt, Reuss, Dilute and the Hashin-Shtrikman Limits allow to calculate the bulk and shear modulus of the material from the percentage of inhomogeneities and Young's modulus and Poisson's coefficient of the materials involved (Gross and Seelig, 2006). According to ASM International (1990) the Young's modulus are 202 GPa and 178 GP, respectively for martensite and bainite. The Poisson coefficient is 0.30 for the two materials.

The purpose of this work was to obtain approximately the bulk and shear modulus of a 4140 steel by different analytical solutions. This material was subjected to quenching treatment in aqueous polymer solution based on polyvinylpyrrolidone at concentrations of 10, 15, 20 and 25% and after quantitative analysis of the resulting phases, it was possible to calculate the two modules under different analytical equations and to compare the obtained results between them.

2. MATERIALS AND METHODS

AISI 4140 cylindrical samples with 25.4mm diameter and 10mm high were prepared for the experiments. These samples were heated for 60 minutes to the temperature of 880°C in resistive oven muffle type, EDG brand, 3000 model. They were then cooled in a tank containing 5000 cm³ of aqueous polymer solution based in polyvinylpyrrolidone (PVP), which was at room temperature. For cooling the pieces were used four different concentrations of polymer solution: 10, 15, 20 and 25%.

After quenching, the samples were sectioned and observed in a light microscope of Olympus brand, model GX 51S, in which images of their microstructure were obtained. With the aim of evaluating the microstructures and different layers of the samples, images were obtained from the center of the piece, from a position close to its surface and an intermediate point between center and surface. These images were evaluated in Image J software, which allows the calculation of the total area of the phases through color differentiation. After obtaining the quantities of phases present in the three points of the material, the phases of intermediate points between these three points were calculated, resulting in eight different points of analysis.

Once the phase quantities were obtained at the different points of analysis of the material and according to the Young's modulus (E) and Poisson's coefficient (υ) of the microstructures found in the samples, the bulk (K) and shear modulus (μ) of the material were calculated analytically, by Equations (1) and (2).

$$K = \frac{E}{3(1 - 2v)}$$
(1)

$$\mu = \frac{E}{2(1 + v)}$$
(2)

Also, using the data from the two different materials, it was possible to calculate the coefficients α and β, which will be used to obtain the properties of the samples, through the analytical calculations. The coefficients α and β were obtained by Equations (3) and (4).

$$\alpha = \frac{1 + v}{3(1 - v)}$$
(3)

$$\beta = \frac{2(4 - 5v)}{15(1 - v)}$$
(4)

The first step in establishing the desired indices was to establish acceptable maximum and minimum limits in the relationship between the matrix and inhomogeneity. These limits are called Hashin-Shtrikman Limits and must be established mathematically by Equations (5) and (6), where "C" represents the percentage of the component in the material, the index "M" is the matrix (Martensite) and the index "I" is the inhomogeneity (Bainite).

$$K_M + \cfrac{C_I}{\cfrac{1}{K_I - K_M} + \cfrac{3.(1 - C_I)}{3.K_M - 4.\mu_M}} \leq K \leq \cfrac{1 - C_I}{\cfrac{1}{K_M - K_I} + \cfrac{3.C_I}{3.K_I - 4.\mu_I}} + K_I \tag{5}$$

$$\mu_M + \cfrac{C_I}{\cfrac{1}{\mu_I - \mu_M} + \cfrac{6.(1 - C_I).(K_M + 2\mu_M)}{5\mu_M(3.K_1 + 4.\mu_M)}} \leq \mu \leq \cfrac{1 - C_I}{\cfrac{1}{\mu_M - \mu_I} + \cfrac{6.C_I.(K_I + 2.\mu_I)}{5.\mu_I.(3.K_I + 4.\mu_I)}} + \mu_I \tag{6}$$

After establishing the Hashin-Shtrikman Limits, the bulk modulus and the shear modulus were calculated for each point of the material under analysis. The four analytical methods selected for analysis were: Voigt, Reuss, Dilute and Mori-Tanaka. The compressibility modulus was calculated respectively by Equations (7), (8), (9) and (10).

$$K_{Voigt} = \sum_{\alpha=1}^{\alpha} C_\alpha K_\alpha \tag{7}$$

$$K_{Reuss}^{-1} = \sum_{\alpha=1}^{\alpha} \frac{C_\alpha}{K_\alpha} \tag{8}$$

$$K_{Dilute} = K_M + C_I . \frac{(K_I - K_M).K_M}{K_M + \alpha.(K_I - K_M)} \tag{9}$$

$$K_{Mori-Tanaka} = K_M + C_I . \frac{(K_I - K_M).K_M}{K_M + \alpha.(1 - C_I).(K_I - K_M)} \tag{10}$$

In turn, the shear modulus is calculated analytically by the methods of Voigt, Reuss, Dilute and Mori-Tanaka using Equations (11), (12), (13) and (14).

$$\mu_{Voigt} = \sum_{\alpha=1}^{\alpha} C_\alpha K_\alpha \tag{11}$$

$$\mu_{Reuss}^{-1} = \sum_{\alpha=1}^{\alpha} \frac{C_\alpha}{K_\alpha} \tag{12}$$

$$\mu_{Dilute} = \mu_M + C_I . \frac{(\mu_I - \mu_M).\mu_M}{\mu_M + \beta.(\mu_I - \mu_M)} \tag{13}$$

$$\mu_{Mori-Tanaka} = \mu_M + C_I . \frac{(\mu_I - \mu_M).\mu_M}{\mu_M + \beta.(1 - C_I).(\mu_I - \mu_M)} \tag{14}$$

Finally, the bulk and the shear modulus were duly calculated for all the points under analysis, and the results obtained were plotted with a view to whether the obtained results respected the Hashin-Shtrikman Limits. Also, the graphics, make the comparison between the results of each calculation analytical methods discussed in the work.

3. RESULTS

The microstructural analysis of the materials demonstrated the formation of two distinct phases: martensite and bainite. The martensite was found in greater quantity, forming the matrix of steel. The bainite, on the other hand, was in smaller quantity, being dispersed in the matrix. Quantities are shown in Table 1.

| Concentration | Position | AISI 4140 | |
		Martensite (%)	Bainite (%)
10 % PVP	Surface	98,7	1,3
	Intermediate	95,5	4,5
	Center	92,8	7,2
15 % PVP	Surface	92,3	7,7
	Intermediate	86	14
	Center	81,5	18,5
20% PVP	Surface	92,1	7,9
	Intermediate	80,2	19,8
	Center	79,1	20,9
25% PVP	Surface	90,8	9,2
	Intermediate	79,1	20,9
	Center	67,5	32,5

Table 1. Quantity of microstructural phases according to the position in the sample of AISI 4140 steel and concentration of the quenching solution

In order to perform the analytical calculations, the martensite was considered the matrix and the bainite the inhomogeneity. Then, the α and β constants were calculated, aiming at the subsequent calculation of the modules by the analytical methods. Using equations (3) and (4), the α = 0.62 and β = 0.476 were calculated.

After that, the Hashin-Shtrikman Limits were calculated using equations (5) and (6) for each of the sample concentrations and positions. Once the limits were established, the bulk and shear modulus were calculated by the Voigt, Reuss, Dilute and Mori-Tanaka methods.

Finally, the results obtained in each of the analytical methods, PVP concentrations and observation points of the samples were plotted comparatively. The bulk modules were calculated and their results are plotted for each situation, as shown in Figure 1:
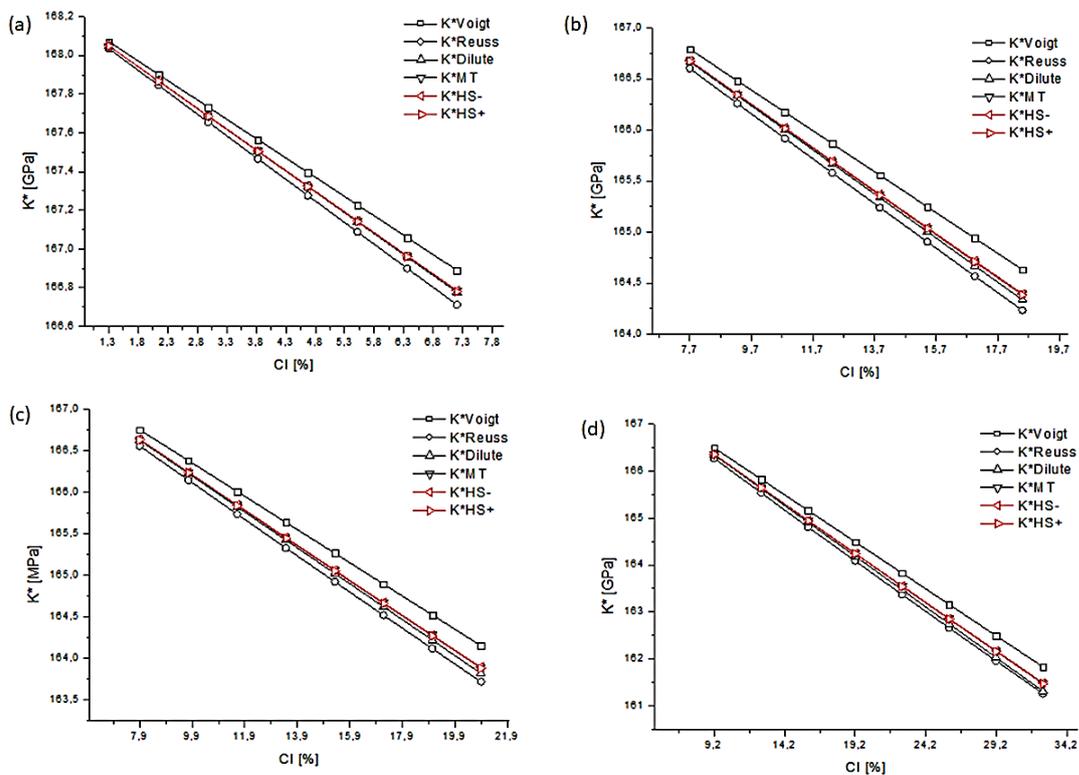

Figure 1. Analytical curves of bulk modulus of different concentrations of aqueous polymer solution. a) 10% b) 15% c) 20% d) 25%

From the observation of the graphs from bulk modulus, it can be affirmed that all the analyzed methods present a similar behavior. In this way, whenever inhomogeneity increases, the curves tend to move away, increasing the difference of their mechanical behavior. The boundaries of Hashin-Shtrikman (HS) are very close, practically overlapping. It is important to highlight that the Mori-Tanaka model follows the behavior of HS, since the HS+ limit used is the Mori-Tanaka model. Additionally, all curves are internal to the limits Voigt and Reuss. The Dilute model has a different behavior, always being below HS and approaching Reuss as the concentration of inhomogeneities increases. In turn, the shear modulus was calculated, resulting in Figure 2:
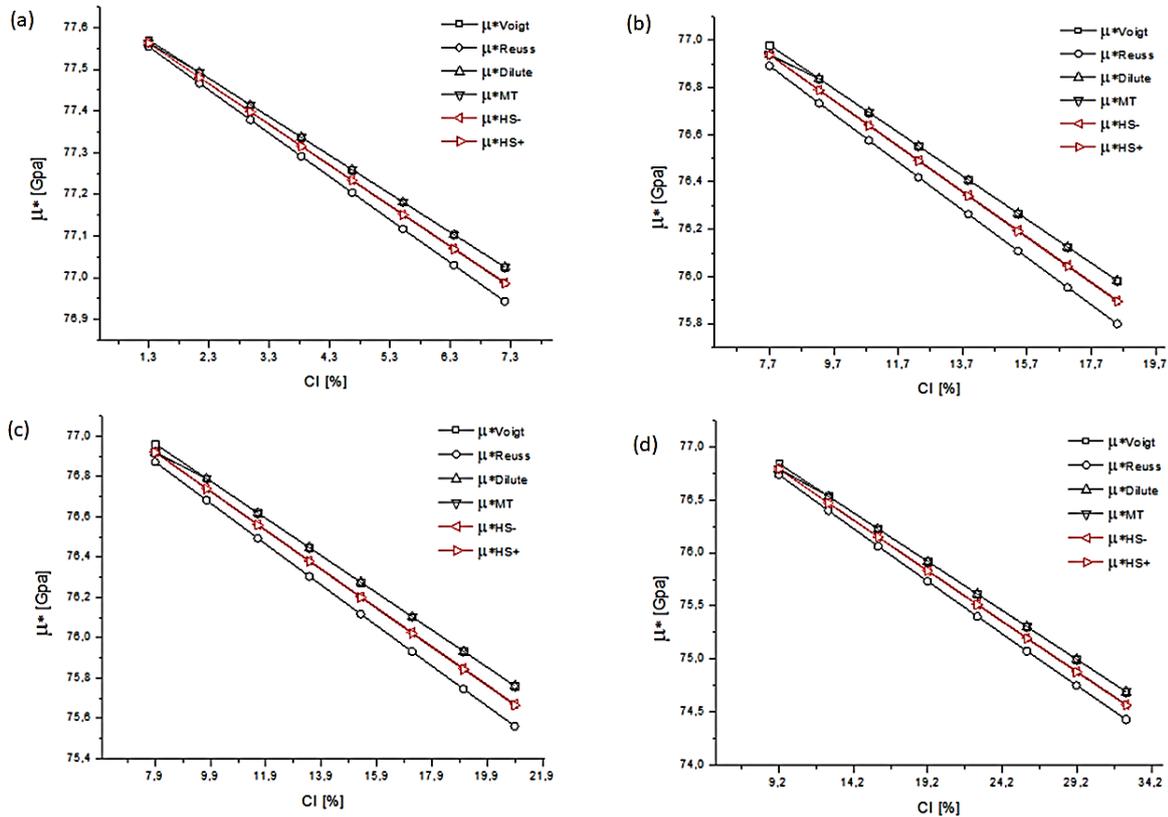

Figure 2. Analytical curves of shear modulus of different concentrations of aqueous polymer solution. a) 10% b) 15% c) 20% d) 25%

As in the transversal elastic modulus, the analytical methods have similar behavior. The HS limits remain very close, almost overlapping and Mori-Tanaka method also behaves superimposed on the HS limits.

The curves are also within the limits of Voigt and Reuss. The Dilute model presented behavior that differs from previous models. Now it develops above HS limits and at low concentrations, it is close to Mori-Tanaka and HS, since with increasing concentration tends to be superimposed on the Voigt model.

4. CONCLUSIONS

The behavior of the curves representing the variation of the bulk and shear modules is practically the same, demonstrating that for these materials and concentrations, there is no significant difference of the modules. This is because, although they are not equal, the Young's modules of the matrix and inhomogeneity are quite close.

This proximity of Young's modulus also results in very close analytic curves and practically superimposed HS boundaries.

Finally, the relation between martensite and bainite in the concentrations of inhomogeneities of this work demonstrates that there is no internal tension originating from the inhomogeneities, because in this case the matrix is harder than the inhomogeneities.

5. REFERENCES

ASM International. "*ASM Handbook: Heat Treatment*". V.4. 10 ed. Russell Township: ASM International, 1990.

Gross, D.; Seelig, T. "*Fracture Mechanics with an Introduction to Micromechanics*", New York, Springer, 2006.

Grum, J.; Bozic, S., "Influence of steel masses and quenchants on mechanical properties of steel", *International Journal of Materials and Product Technology*, v. 24, pp. 224–240, Oct. 2005.

Koudil, Z.; Ikkene, R.; Mouzali, M., "Cooling capacity optimization: calculation of hardening power of aqueous solution based on poly(n-vinyl-2-pyrrolidone)", *Journal of Materials Engineering and Performance*, v. 23, pp. 551-559, Dec. 2014.

Heming, C.; Xieqing, H.; Jiabin, X., "Comparison of surface heat-transfer coefficients between various diameter cylinders during rapid cooling", *Journal of Materials Processing Technology*, v. 138, pp. 399–402, Jul. 2003.

Hernandez, H.J.V.; Morales, B.H., "A novel probe design to study wetting front kinematics during forced convective quenching", *Experimental Thermal and Fluid Science*, v. 33, pp. 797–807, Jul. 2009.

Magee, C.L.; Davies, R.G., "The volume expansion accompanying the F.C.C. to B.C.C. transformation in ferrous alloys", *Acta Metallurgica,* v. 20, pp. 1031-1043, Aug. 1972.

Ramesh, G.; Prabhu, K.N., "Effect of polymer concentration on wetting and cooling performance during immersion quenching", *Metallurgical and Materials Transactions B*, v. 47, pp. 859-881, Dec. 2016.

Totten, G.E., "*Steel heat treatment handbook"*, 2 ed., New York, CRC Press, 2007.

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