ORIGINAL PAPER

STUDY OF THE MECHANICAL AND STRUCTURAL PROPERTIES OF HASTELLOY C2000

A. AMRI[1,2], O. BENLENDA[2], F. SABIR[2], L. ZERROUK[2], A. IBNLFASSI[1], E. SAAD[2]

Manuscript received: 26.10.2016; Accepted paper: 11.11.2016;
Published online: 30.12.2016.

Abstract. *Hastelloy C2000 is a nickel-base superalloy. With the addition of copper to nickel-chromium-molybdenum system, it is resistant to a wide range of corrosive chemicals as well as the combination of molybdenum and copper creates an excellent corrosion resistance to reducing media, while the high content of the chrome provides a good oxidation resistance. Its structure is face-centered cubic.*

The value of our work is to study the stability of the Hastelloy C2000 on its microstructure and mechanical properties and this by a heat treatment at four temperatures: 700 °C, 800 °C, 900 °C and 1000 °C with a prolonged maintenance at each temperature for three hours. The monitoring of these two aspects is performed using the following experimentals techniques: optical microscopy and hardness measurement.

After heat treatment, the pictures obtained by optical microscopy revealed the presence of different morphologies of carbides, joints and inside the grains, also we remark the appearance of the discontinuous transformations and the coalescence phenomenon of grain to form a larger area of precipitation. For hardness in the over-aging, it attains a final value of 47 for the four samples treated at 700 °C, 800°C, 900 °C and 1000 °C.

Keywords: *Hastelloy C2000, Heat treatment, hardness, optical microscopy.*

1. INTRODUCTION

The choice of a material for a given application requires precise and reliable knowledge of durability, performance and stability in conditions of employment, especially environmental and this more important for alloys intended to work on high temperatures.

Nowadays, the austenitic stainless steel is essentially used on the industrial domain as the piping and the equipment for chemicals [1]. In most of these applications the mechanical properties of these steels are satisfactory and do not require improvement. However, in certain cases where this type of material is used in devices operating in high temperatures, these steels are not usable, either because of their insufficient mechanical and microstructural properties, or because of their sensibility and instability with certain ways of functioning.

In the case of more aggressive environments at high temperatures, it is the Ni-based superalloys, which are used, for example for the aeronautical and land-based turbines. Indeed, these metallic materials combine high mechanical characteristics in a wide domain of temperatures and an excellent resistance to oxidation and corrosion [1].

[1] University Hassan 1st, FSTS, Laboratory of Environmental Sciences and Development, Settat, Morocco.
E-mail : saadelmadani73@gmail.com.
[2] University Hassan 1st, FSTS, Laboratory of Physical Chemistry of Process and Materials (PCPM), Settat, Morocco.

To answer partially in this problem, numerous research were led, we notice the study on alloys with nickel: microstructure and mechanical properties of Jean Yves Guédou [2], and the study of the structural modifications of superalloys with nickel of Sébastien Chollet's [1]. In particular, we find studies on the effects of heats treatments on the microstructure of alloys [3-5].

It is in the same context as joins our work, which consists to study the influence of heats treatments in high temperatures on the structural stability and on the mechanical properties as being key points for the choice of materials for the various applications.

Among the different shades of austenitic stainless steels based on nickel, we differentiate the HASTELLOY C2000 which makes the object of our study. This alloy contains a nominal quantity of 52-60 % of Ni, 15-31 % of Cr, from 9 to 16 % of Mo, and small additions of others elements such as the iron, the copper, the tungsten, etc [6]. This alloy has a face-centered cubic structure. Because of its structure, it has an excellent ductility, good malleability and it is easily weldable [7].

2. MATERIALS AND METHODS

2.1. PREPARATION OF SAMPLES AND COMPOSITION

In our study we used a plate given by Haynes International Inc. Small samples of dimensions (18mm x 16mm x 3mm) were cut up with metallic saw.
The composition of the Hastelloy C2000, which was the object of the metallurgical study, is given in the table below [8]:

Table 1. Chemical composition of Hastelloy C2000.

Ni[a]	Cr	Mo	Cu	Fe	Co	Al	Mn	C	Others
59	20 - 24	15 – 17	1,3-1,9	3,00**	2**	0,5**	0,05**	0,01**	P = 0,025** Si = 0,08** Sulfides = 0,01**

** : Maximum percentage, a : balance

According to the diagram of ternary phase Ni-Cr-Mo [9] presented below for the temperatures 600 °C, 850 °C and 1000 °C ,and with the proportions of our alloy which are 52 to 60 % of Ni, 15 to 31 % of Cr and from 9 to 16 % Mo, we can remark that the alloy has an austenitic structure for the various temperatures of treatment.

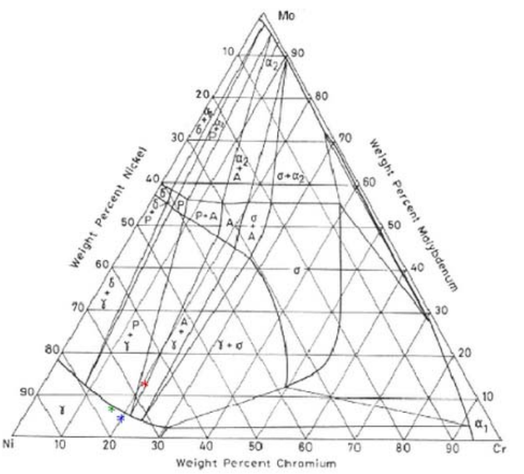


Figure 1. Phase diagram Ni-Cr-Mo at 600 °C.

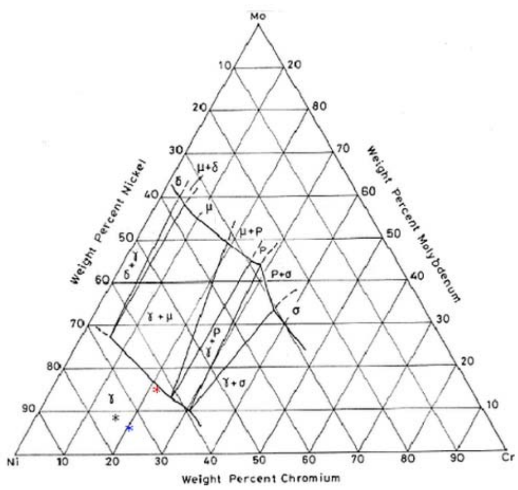


Figure 2. Phase diagram Ni-Cr-Mo at 850 °C.

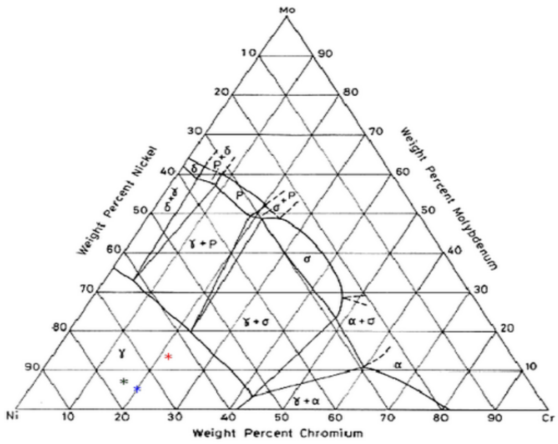


Figure 3. Phase diagram Ni-Cr-Mo at 1000°C.

We also notice that by increasing the temperature, and by increasing the content of the nickel, the domain of the austenite widened while if we increase the proportion of chromium, we favor the precipitation of the phase alpha.

2.2. HARDNESS

The measure of the hardness of the alloy C2000 is a way to follow the mechanical behavior of this alloy and its aging rate.

Hardness tests are performed by the ROCKWELL method F (HRF), using a universal hardness tester. The indenter used is steel ball type with 1.588 mm in diameter under a load of 60 kgf. Each measurement is the average of four marks distributed on the flat surface of the sample, obtained by mechanical polishing under water to avoid overheating which can cause changes in the structure and on abrasive paper with decreasing size grading turning around a fixed axis.

2.3. OPTICAL MICROSCOPY AND SCANNING ELECTRON MICROSCOPY (SEM)

In order to detect changes in the structure, we used optical microscopy and scanning electron microscopy. The samples require mechanical polishing followed by the revelation of the surface by means of etching using aqua regia, which consists of a mixture of hydrochloric acid and concentrated nitric acid in a proportion of 1 volume of nitric acid for 3 volumes of hydrochloric acid [10].

3. RESULTS AND DISCUSSION

3.1. STUDY OF HASTELLOY C2000 AT 800 °C

3.1.1. HARDNESS MEASUREMENT

According to the Fig. 4, we notice that for the sample treated at 800 °C, the hardness gradually decreases from its initial value that is 50 to 46, 21 days after quenching, before settling at the final value wich is 47.

We also note that the kinetic of the ageing is much faster than we were not able to visualize it, contrary to the kinetic of the over-ageing, which is slow.This drop in hardness was also observed for other alloys based on nickel and is probably due to the formation of precipitates stable at high temperatures [11].

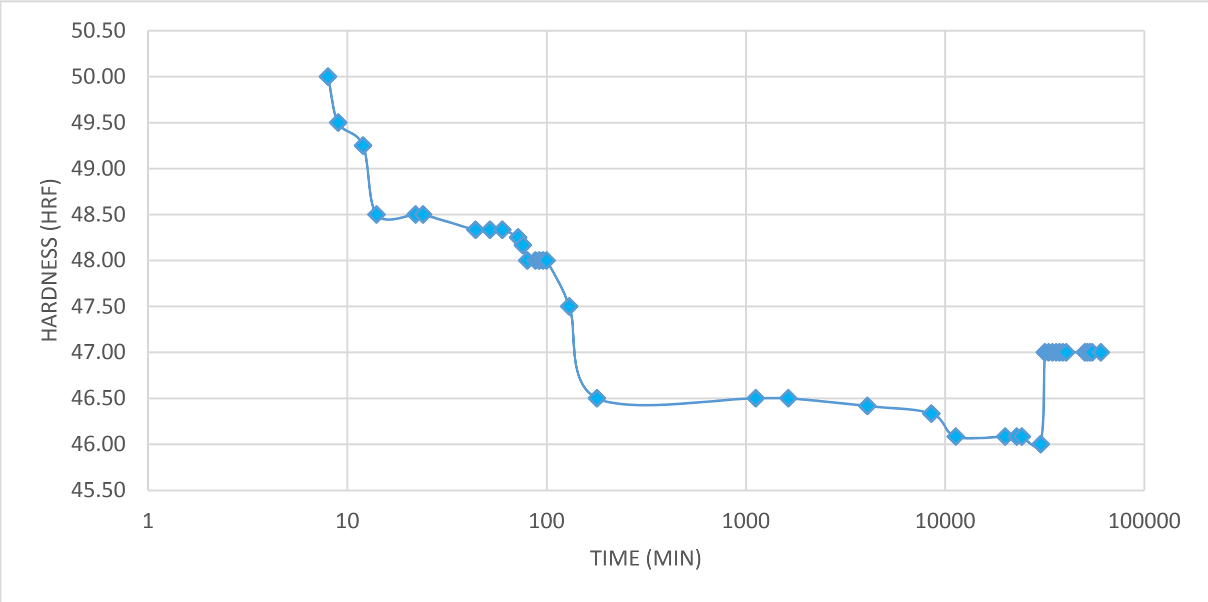


Figure 4. Evolution of hardness in HRF of piece treated at 800°C as function of time.

3.1.2. MICROSCOPIC STUDY

For pictures taken in the optical microscope, they show that, 100 min after quenching, we obtain the appearance of some small grains dispersed in the matrix. We also find, 24 hours after quenching of the same alloy treated at 800°C (Fig. 5), a set of discontinuous transformations distributed on the matrix what characterizes the phase of the ageing of the alloy.

After 48 hours of quenching (figure 6), we begin to observe a precipitations which are accelerated over 550 °C and which usually begin on the grains boundaries where the diffusion is much faster than inside the matrix and it is for this part that the interfacial energy is more important [12]. However, these precipitations form a continuous film on the grain boundary, which leads to a degradation of the mechanical properties of the material [6].

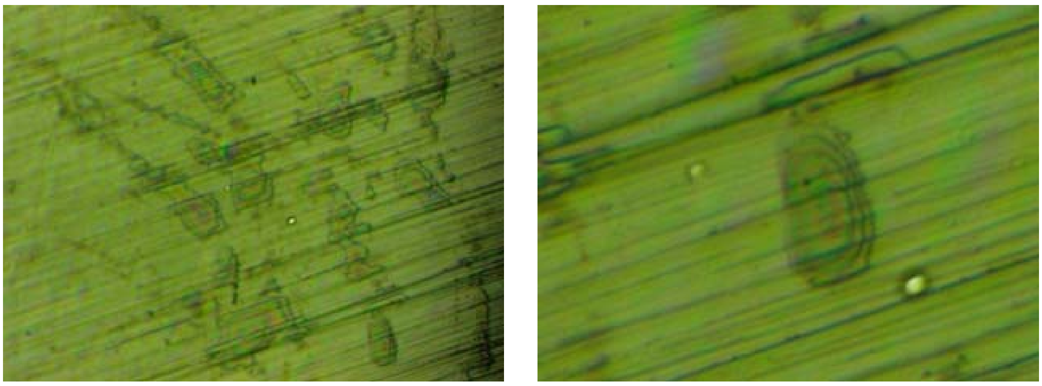


Figure 5.Visualization of the structure of C2000 treated at 800 °C, attacked with aqua regia, 24 hours after quenching.

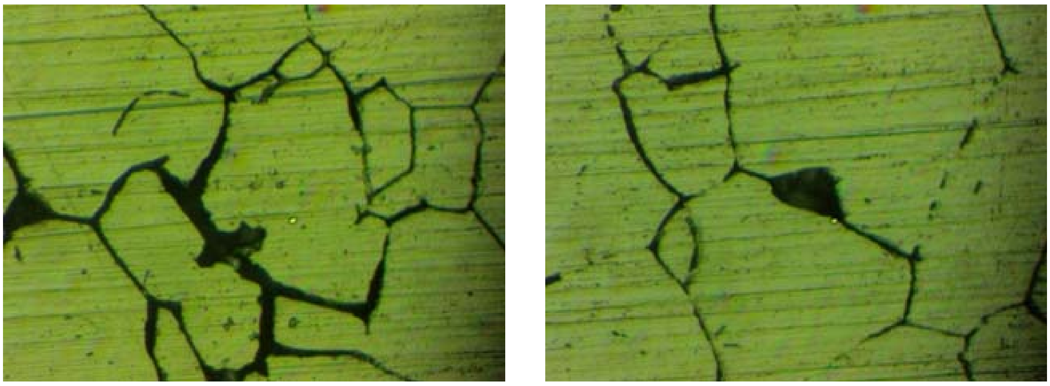


Figure 6.Visualization of the structure of C2000 treated at 800 °C, attacked with aqua regia, 48 hours after quenching.

Fig. 7 shows the formation of intergranular and intragranular carbides after 29 days of quenching, which have generally globular form, with the appearance of discontinuous precipitations within the grains. This discontinuous carbides precipitations often leads to local impoverishment of Cr in the grain boundary of the surrounding matrix and raising the grain boundary to intergranular oxidation [13]. In the same figure, we remark that the matrix is covered by grains of different sizes. In addition, we remark the phenomenon of the coalescence of grains, which leads to form larger ones.

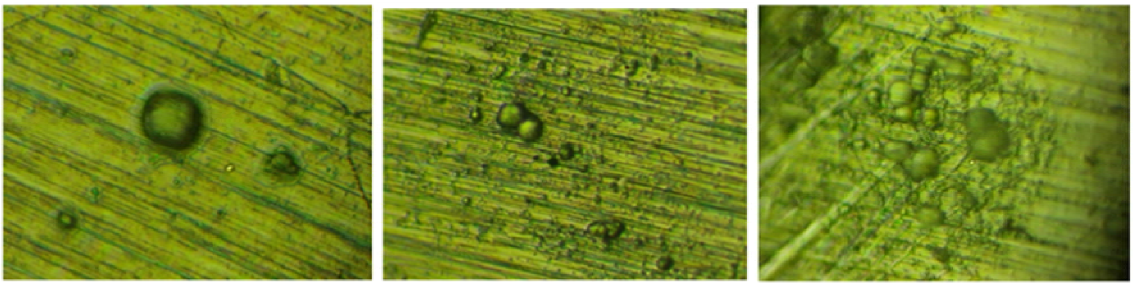
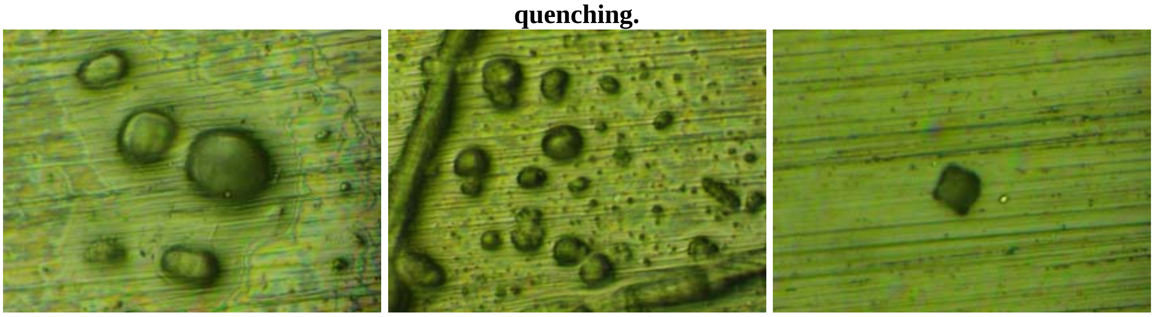


Figure 7.Visualization of the structure of C2000 treated at 800 °C, attacked with aqua regia, 29 days after quenching.



(a) (b) (c)

Figure 8. Visualization of the structure of C2000 treated at 800 °C, attacked with aqua regia, 38 days after quenching.

Fig. 8, we note that the matrix is covered with very large areas of precipitation of carbides in different morphologies; these areas are also surrounded by discontinuous precipitations with a movement of the fronts of these precipitates, which is also observed in other alloys nickel base treated at the same temperature [14].In the same figure, we notice the appearance of a new form of precipitation that is the square shape after 38 days of quenching (Fig. 8 c).

3.2. STUDY OF HASTELLOY C2000 AT 1000°C

3.2.1. HARDNESS MEASUREMENT

From the Fig. 9, the hardness remains almost constant in the first 30 minutes after quenching, and it increases almost linearly until the maximum value, which is in the range of 50.33 after 44 minutes. Then the hardness decreases slowly until the value 46,5 after 6 days before stabilizing in the final value, which is 47. From the curve of hardness, we also notice that the kinetic of the ageing is faster contrary that of the over-ageing which is much slower.

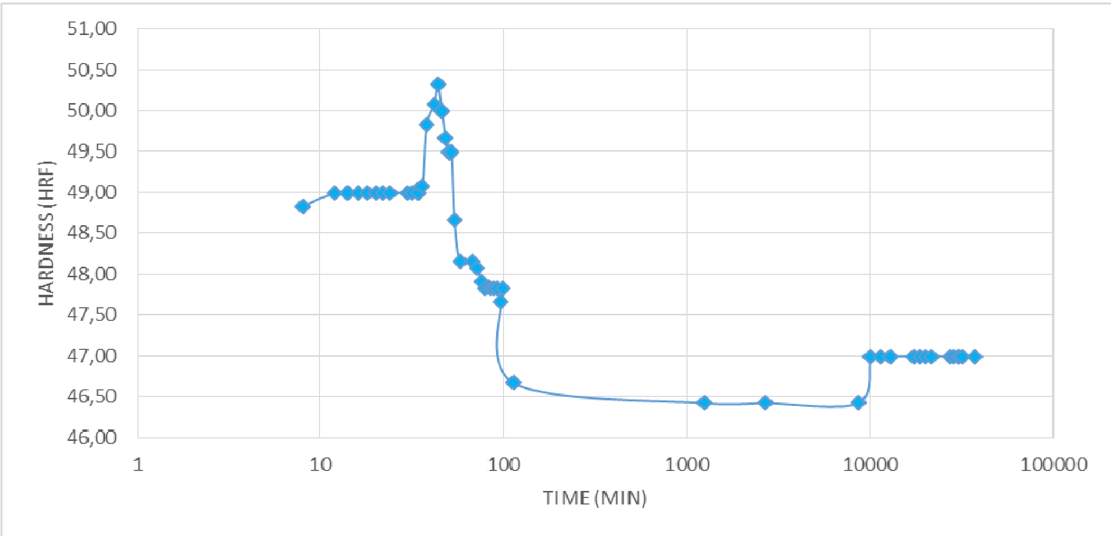
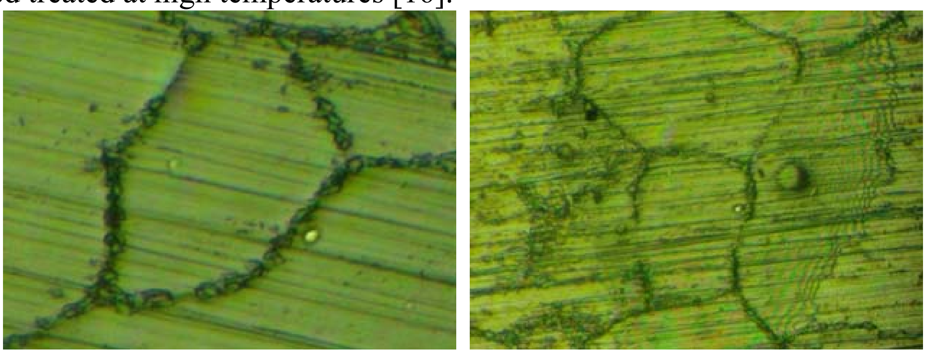


Figure 9. Evolution of hardness in HRF of piece treated at 800°C as function of time.

3.2.2. MICROSCOPIC STUDY

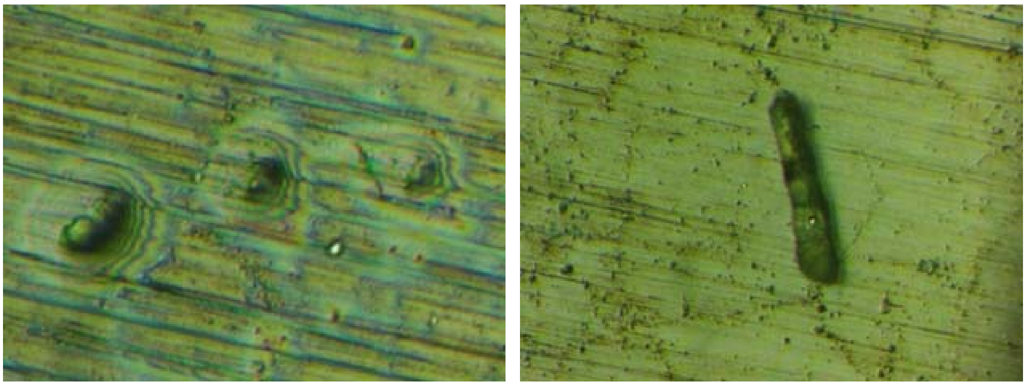
For the sample treated at 1000 °C, the precipitates carbides precipitate in excess at grains boundaries 100 minutes after quenching (Fig. 10a) because at high temperatures, chromium and carbon diffuse into grains boundaries to form chromium carbides. In these areas, the content in chromium is widely superior compared to the rest of the alloy and this can cause the corrosion of these areas with low content in chromium [15].

We also note, that after 24 hours of quenching (Fig. 10b), we begin to see the intergranular and intragranular precipitations in granular and polyhedral shape. This is accompanied by the large areas of discontinuous transformations that characterize the stage of aging and what suits to the results of the hardness.This is visualized for other alloys with nickel-based treated at high temperatures [16].



(a) **(b)**

Figure 10. Visualization of the structure of C2000 treated at 1000 °C, attacked with aqua regia, 100 minutes and 24 hours after quenching.

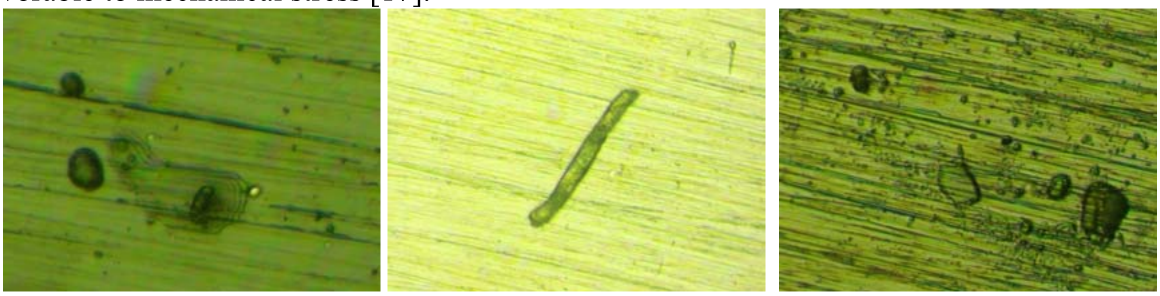


(a) (b)

Figure 11. Visualization of the structure of C2000 treated at 1000°C, attacked with aqua regia, 14 days after quenching.

The Fig. 12 shows, after 15 days of quenching, that the matrix is covered with the areas of precipitation of grains and in the same figure, we remark the phenomenon of coalescence between the various grains of various morphologies which will lead to more precipitation extended over the whole of the matrix. We also note the emergence of a new form, which is lamellar form (Figure 12, b), and discontinuous precipitations of grains.

After 20 days of quenching, we observe that the grains continue to grow and to propagate throughout the matrix with intergranular and intragranular precipitations.
We also note that the size of grains influences significantly the properties of alloys, because in the case of a fine grain structure is more resilient than a large grain structure, which is not favorable to mechanical stress [17].



(a) (b) (c)

Figure 12. Visualization of the structure of C2000 treated at 1000°C, attacked with aqua regia, 22 days after quenching.

3.3. INFLUENCE OF HEAT TREATMENT

3.3.1. STUDY OF HARDNESS

The Fig. 13 shows the evolution of the hardness for the four samples of C2000 alloy, treated at 700°C, 800°C, 900 °C and 1000 °C. We note that, for the samples treated at 700°C and 1000°C, the kinetics of aging are faster compared to kinetics of the over-aging. While for samples treated at 800°C and 900 °C, the speed of the kinetics of aging prevented us to see the transformations immediately after quenching.

- For the sample treated at 700 °C, the hardness remains constant in the first 12 minutes before increasing to 50.5, which is the maximum value after 16 minutes. After that, the hardness gradually decreases during the phase aging to 46, before stabilizing at the final value, which is 47 after days 35.

- For the sample treated at 800°C, it is noted that the hardness begins to decrease an almost linearly from the maximum 50, which is almost the same comparing the sample treated at 700°C, until the value 48.5 in the first 14 minutes. After it continues to decrease gradually to the value 46 before stabilizing at 47 after 42 days.
- For the sample treated at 900°C, the hardness decreases slowly to the maximum value which is 47 until 46, before recovering and stabilizing at the final value 47, after 35 days, which is similar compared to others samples.
- For the sample treated at 1000°C, we notice that the hardness remains almost constant during the first 34 minute, before increasing until the maximal value 50,33 then it decreases significantly until 46,5, before stabilizing in the value 47 after 30 days.

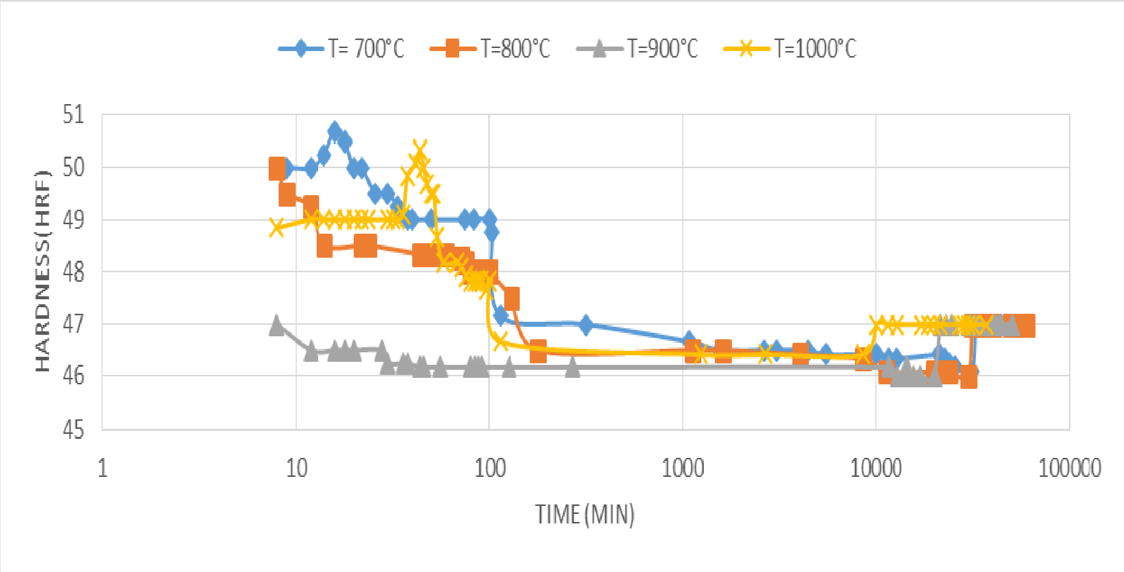
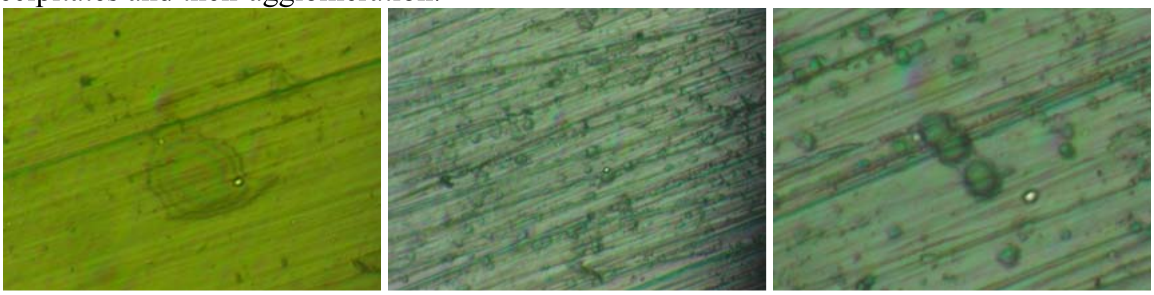


Figure 13. Evolution of hardness in HRF of pieces treated at 700°C, 800°C, 900°C and 1000°C as function of time.

3.3.2. MICROSCOPIC STUDY

According to the Fig. 14, we note, after 48 hours of quenching of the alloy C2000 treated at 700°C, the appearance of the grains boundaries throughout the matrix and discontinuous transformations. We remark also the formation of intergranular precipitates with different sizes.

For the sample treated at 900°C (Fig. 14b), after almost the same quenching time, the matrix is covered with precipitates with different morphologies: globular and polyhedral. In the same figure, we begin to show the phenomenon of coalescence of grains (Fig. 14c). Therefore, we can say that an increase of the temperature favors the formation of precipitates and their agglomeration.

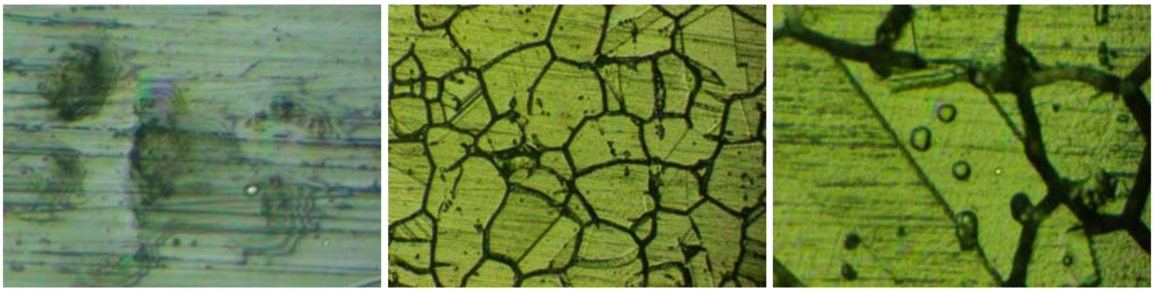


(a) (b) (c)

Figure 14.Visualization of the structure of C2000 treated at 700 °C and 900 °C, attacked with aqua regia, 48 hours after quenching.

For the sample treated at 700 °C, after 7 days of quenching, we have the appearance of large areas of discontinuous precipitations of grains (Figure 15 a). Moreover, for the sample treated at 900°C, after the same time of quenching, we remark the presence of primary precipitations in the grains boundaries, which lead to form a continuous film. We have also a fine particle that dispersed inside the matrix. In addition, in the same figure, we note the appearance of intragranular precipitations and the presence of aureoles precipitations dispersed in the matrix (Fig. 15, b and c).

This kind of precipitate characterizes this alloy treated at high temperatures for a prolonged maintenance, and this is met for the G35 alloy nickel-based treated at the same temperature [16].

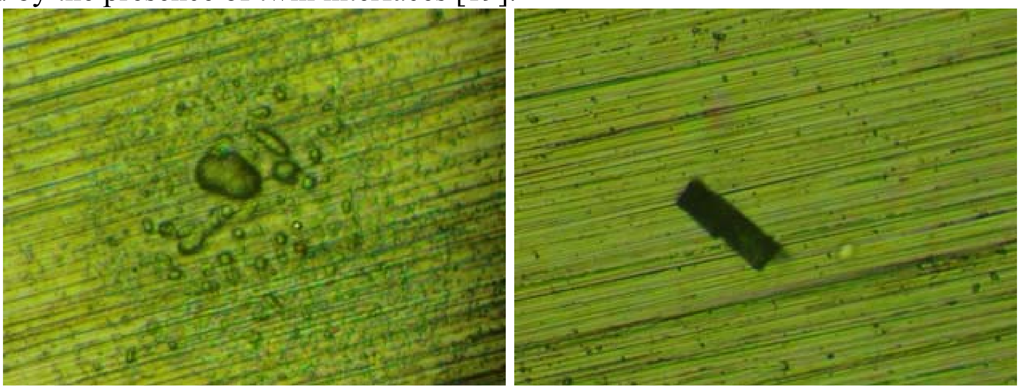


(a) (b) (c)

Figure 15.Visualization of the structure of C2000 treated at 700°C and 900°C, attacked with aqua regia, 7 days after quenching.

The Fig. 16 shows a large area of precipitation with the presence of different morphologies and different sizes. In the same figure, we remark the emergence of a new form that is lamellar shape, after 28 days of quenching, for the sample treated at 700 °C. After almost the same amount of the quenching process, which is 29 days for the sample treated at 900 °C (Fig. 17), we note a growth of intergranulars and intragranulars grains, and some areas transformed by the discontinuous magnification which is responsible for over-aging and which is manifested by a reaction mechanism with coarse and incomplete lamellar precipitation. In general, this reaction is characterized by a lamellar texture localized near to the grains boundaries [18].

We also note the presence of the form "Twin" as one of the types of deformations dislocation, and this affects properties of a variety of material, including such technologically important material as superalloys, whose fatigue behavior at high temperature is drastically affected by the presence of twin interfaces [19].



(a) (b)

Figure 16. Visualization of the structure of C2000 treated at 700°C, attacked with aqua regia, 28 days after quenching.

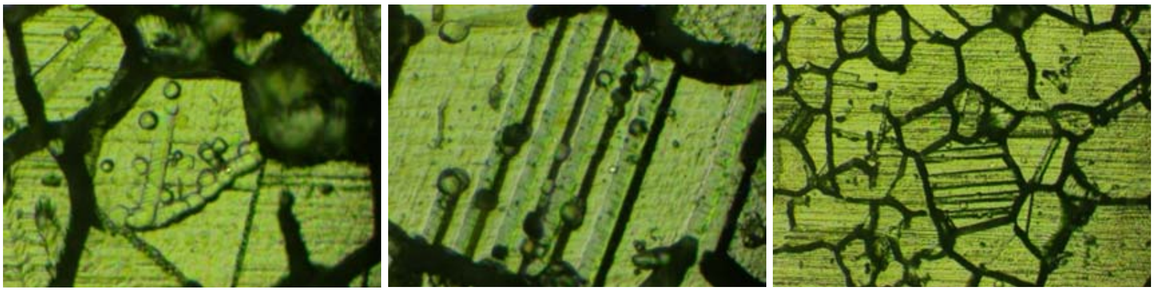

(a) (b) (c)

Figure 17. Visualization of the structure of C2000 treated at 900°C, attacked with aqua regia, 29 days after quenching.

3.4. THE KINETIC STUDY OF THE TRANSFORMATION OF THE HASTELLOY C2000 DURING THE OVER-AGING

3.4.1. APPLICATION OF EQUATION OF MEHL AND JOHNSON

We study the kinetic of transformation during the over-aging of the HASTELLOY C2000 from the hardness measurements for two months. We used the equation of Johnson-Mehl-Avrami [20] to determine the index n and the constant K. The heat treatments were realized at temperatures of 700 °C, 800 °C, 900 °C and 1000 °C during 3 hours.

The matter content changed in function of time at a given temperature is summarized in the following equation:

$$y = 1 - \exp\left(-Kt^{n}\right) \qquad (1)$$

which can also be written as:

$$\ln(-\ln(1 - y)) = n \ln(t) + \ln(K) \qquad (2)$$

The degree of advancement x is calculated from the following relation*:*

$$x = \frac{HRF\,(t) - HRF\,(0)}{HRF\,(\infty) - HRF\,(0)} \qquad (3)$$

with: HRF (t): Hardness at time t;
HRF (0): Maximum hardness;
HRF (∞): Final hardness.

To determine the constants n and K, we plot ln (-ln (1-x)) as a function of ln (t) (Fig. 18). The linear regression of data points obtained gives a slope n and x-ordinate ln (k). The table 2 gives the values of the exponent n and the rate constant K for different temperatures.

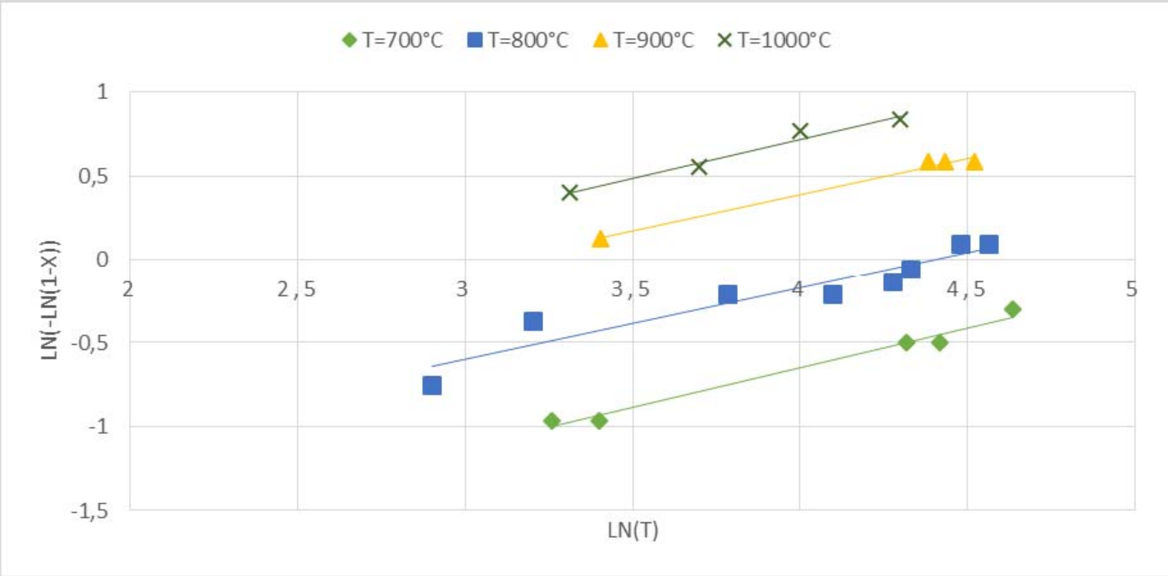


Figure 18. Representation of (-ln (1-x)) versus ln (t) during the overaging of HASTELLOY C2000 treated at 700 °C, 800 °C, 900 °C, and 1000 °C.

Table. 2. Coefficients of the equation and Johnson Mehl-Avrami of HASTELLOY C2000 treated at 700 °C, 800 °C, 900 °C and 1000 °C for 3 hours and quenched with water.

T (°C)	n	K (min^{-1})
700	0,4717	$7{,}91.10^{-2}$
800	0,4253	$1{,}54.10^{-1}$
900	0,4363	$2{,}57.10^{-1}$
1000	0,4647	$3{,}20.10^{-1}$

From the table 1, we remark that the coefficient n is almost the same for the four temperatures 700 °C, 800 °C, 900 °C and 1000 °C. From this value of n, we can deduce that the reaction mechanism of precipitation softening is simple. In addition, we note that by increasing the temperature the value of K increases.

3.4.2. DETERMINATION OF THE APPARENT ACTIVATION ENERGY

To determine the apparent activation energy Q, we used the method of bruke [20]. This method consists in drawing for different values of the advancement x, ln (t) versus 1/T (Fig. 19) which gives a slope of straight line Q / R where R is the perfect gas constant and equals to $8{,}32 J.mol^{-1}.K^{-1}$.

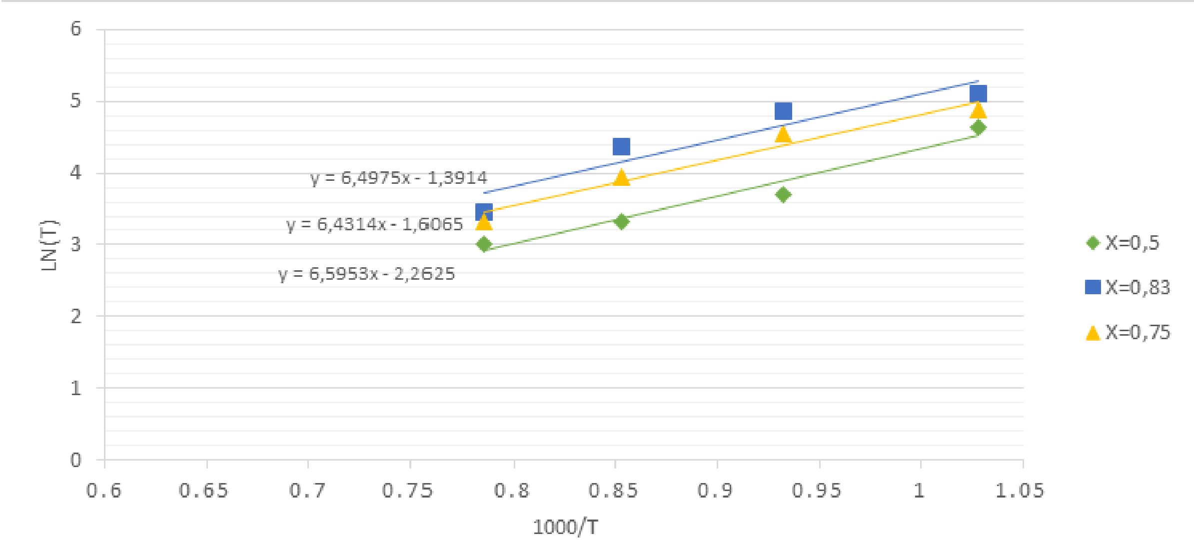


Figure 19. Representation of ln(t) versus 1000/T of HASTELLOY C2000 treated at 700 °C, 800 °C, 900 °C and 1000 °C.

The table 3 shows the different values of Q. The average value of the apparent activation energy according to the method of Bruke for X= 0,5, X= 0,83 and X=0,75 is **54.1470 KJ/mol.**

Table. 3. Values of Apparent activation energy Q for diffent values of X.

X	Apparent activation energy Q in (KJ/mol)
0,5	54,8728
0,83	54,0592
0,75	53,5092

From the table 3, we observe that the apparent activation energy Q remains almost constant for different values of degree of advancement X, therefore we can conclude that Q is independent of X.

To verify the value of the apparent activation energy Q, found by the method of Bruke, we will use the values of K previously found to recalculate the value of Q based on the Arrhenius relation [21]:

$$\log\left(\frac{K1}{K2}\right) = -\frac{Q}{R}\left(\frac{1}{T1} - \frac{1}{T2}\right) \quad (4)$$

The table 4 shows the values of the apparent activation energy found by the Arrhenius relation. The average of the apparent activation energy found by this method is **53.2593 KJ/mol.** By comparing the two values found by the method of Bruke and by the Arrhenius relation, we note that they are almost similar.

Table. 4. Values of the apparent activation energy obtained by the Arrhenius relation.

T1 (°C)	T2 (°C)	Q (KJ/mol)
700	**800**	57,6858
800	**900**	54,0658
700	**1000**	48,0265

4. CONCLUSIONS

In this work, we treated the superalloy C2000 for 3 hours at temperatures of 700 °C, 800 °C, 900 °C and 1000 °C. These heats treatments resulted in a final hardness, which is equal to 47 for four processing temperatures.

For the microscopic study after quenching, it showed the presence of intraganulars and intergranulars precipitations of carbides with different morphologies: globular, polyhedral, lamellar and square. As was visualized we have also a large discontinuous precipitation areas that characterize the phase of over-aging of the hastelloy C2000 and coalescence of different grains to form other with a larger sizes and this is consistent with the results of hardness.

According to the equation of JOHNSON-MEHL and the equation of Avrami, we note that the structural transformations are performed with a kinetic of 0,45 indicating that the reaction mechanism of precipitation softening is simple and requires an average activation energy of 53,7031 KJ/mol determined by the method of Bruke and the Arrhenius equation.

REFERENCES

[1] Chollet, S., *Étude des modifications microstructurales de superalliages à base nickel induites par nitruration assistée plasma,* University of Poitier, SIMMEA, Science and Engineering Materials, Mechanics, Energy and Aeronautics, 2014.

[2] Guédou, J.Y., Revaud, M., Devaux, A., Dumont, A., *Superalliages à base nickel : microstructures et propriétés mécaniques,* Seminar November 23, Materials Centre, Evry, 2012.

[3] Furrer, D.U., *A review of U720 LI alloy and process development*, in Materials Design Approaches and Experiences, p: 281-296, 2001.

[4] Brooks, R.R., Rainforth, W.M., *Fatigue & Fracture of Engineering Materials & Structure*, **23**, 725, 2000.

[5] Monajati, H. et al., *Materials Science & Engineering*, **A373**, 286, 2004.

[6] ASM International Handbook Committee, *Nickel, Cobalt and Their Alloys*, ASM International, United States of America, 2000.

[7] ASM International, *Corrosion in the Petrochemical Industry*, Second Edition, United States of America, December 14, 2015.

[8] Schweitzer, P.A., *Fundamentals of Metallic Corrosion: Atmospheric and Media Corrosion of Metals*, CRC Press 2006.

[9] Talekar, A.S., *Oxidation Behavior of Nickel-base Superalloys and High Strength Low Alloy (HSLA) Steels at Elevated Temperatures*, University of Nevada, Reno. Metalurgical Engineering, August 2008.

[10] ASM International Handbook Committee, *ASM Handbook: Volume 9: Metallography and Microstructures*, USA, 2004.

[11] Sadek, K. et al., *Journal of Advanced Research in Science and Technology*, **2**(2), 238, 2015.

[12] Gharehbaghi, A., *Precipitation Study in a High Temperature Austenitic Stainless Steel using Low Voltage Energy Dispersive X-ray Spectroscopy*, Master Degree Project, 2012.

[13] Benoît, T.O., *Etude comparative de différents superalliages base Ni pour ressorts de systèmes de maintien*, National Polytechnic Institute of Toulouse, 2011.

[14] Ben Lenda, O. et al., *Journal of Science and Arts*, **4**(33), 351, 2015.
[15] Davis, J.R., ASM International, *Corrosion: Understanding the Basics*, United States of America, 2000.
[16] Sabir, F. et al., *Journal of Science and Arts*, **3**(32), 269, 2015.
[17] Schewmon, P.G., *Transformations in metals,* MC.Grow-Hill, New York, 1969.
[18] Zantalla, E. et al., *J. Mater. Environ. Sci*., **7**(6), 2094, 2016.
[19] Rath, B.B. et al., *Mater. Phys. Mech.*, **1**, 61, 2000.
[20] Bruke, J., *The kinetics of phases transformations in metals*, Pergamon Press, New York, 1965.
[21] Ben Lenda, O. et al., *Journal of Science and Arts*, **2**(31), 165, 2015.