



The Effects of Long-Term Operation and High Temperature on Material Properties of Austenitic Stainless Steel Type 321H

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Abstract

Changes in mechanical properties of material as a result of service in different conditions can be provided by mechanical testing to assist the estimation of current internal situation of these materials, or the degree of deterioration may exist in furnaces serviced at high temperature and exceed their design life. Because of the rarity works on austenitic stainless steel material type AISI 321H, in this work, ultimate tensile strength, yield strength, elongation, hardness, and absorbed energy by impact are evaluated based on experimental data obtained from mechanical testing. Samples of tubes are extracted from furnace belong to hydrotreater unit, also samples from un-used tube material are used to make comparisons between these properties. Tensile properties of stainless steel (AISI 321H) were decreased as temperature increases; the trend of properties decreasing for the samples of un-used tube material is the same for the ex-used material. The trend of stress-strain curve will not change due to elevated temperature exposure for long time of service, except the yield strength will be higher in this diagram. The yield strength increased under these conditions, but the ability of material which is elongated will decrease. Hardness and absorbed energy increased by 11.28 and 14% respectively when the material is aged for long time under effect of high temperature accompanied with creep effect.

Keywords: *Hardness, Impact, Mechanical Properties, Stainless Steel 321H, Mechanical Properties, Tensile Strength, Tube Furnace.*

1. Introduction

Austenitic stainless steels are widely used in engineering applications; these types of alloys are selected to serve at high temperature due to their high tensile strength and good creep resistance. Furnaces in reforming units are the core of these units provided by heat to produce hydrogen from hydrocarbons. The nominal life design for such furnaces is 100000hr on the basis of standard API 530[1]. Assessment of damage in these furnaces is an important factor in determining their remaining safe life. Components in service at elevated temperatures for long term can fail due to excessive creep deformation or cracking.

Therefore, improvement of material strength and other properties for these components is the key to resist such failures. The assessment of material properties, such as hardness, impact energy, and tensile strength near the end of design life or beyond that is vital to provide a safe working life for the unit and prevent a catastrophic failure.

Hardness measurements are the best ways to detect if there are metallurgical changes [2,3]. Recent developments in the technique of hardness have shown that it can be used in a predictive sense to estimate the remnant life [4], a hardness based approach is used to evaluate the creep strength of superheater tubes of boilers [5]. The impact test also has been used in testing of steel

products which is related to the behavior of metal, and in some cases, making the tests at properly chosen temperatures other than room temperature [6]. The remaining life of furnaces tubes can be predicted based on fracture toughness and mechanical properties, also a suggestion to pay more attention to avoid the excessive impact during starting up and shutdowns because of the decrease in material ductility [7].

A comparison of both mechanical properties at different temperatures and the roles of these properties to resist creep [8], can be useful to give good indications for the degree of internal damage due to prolonged exposure to temperature, when these comparisons are made between the un-used and ex-used material.

Austenitic Stainless steel AISI 321 is stabilized by titanium addition. This grade of steel is used in engineering applications under high temperature, because of its high strength and good creep resistance. While, stainless steel AISI 321H is modified to stainless steel AISI 321 with higher carbon contains, it was developed to enhance the creep resistance and the higher strength at temperature above 537°C. Many researches cover the stainless steel material type 321 from different viewpoints [9, 10, 11, 12, 13, 14, 15], on the other hand, there is a gap in the published researches about the modified stainless steel material type 321H.

Because of the rare work on austenitic stainless steel material type 321H, the present work investigates a furnace tube (O.D. 141.3 mm, thickness 6.55 mm) made from this type of steel and served in complex reformer/naphtha hydrotreater unit for more than 290000hr, the design tube wall temperature is 570°C. This investigation uses the mechanical properties, such as hardness, impact energy, yield strength, and tensile strength at different temperatures to make a comparison between an ex-used tube with un-used one of the same dimensions and material. Tensile tests were done at four different temperatures 25°C, 300°C, 500°C, and 700°C, while impact tests were conducted at room temperature and at the maximum service temperature 466°C.

2. Experimentation

2.1. Chemical Analysis of Tube Samples

In this work, two different ages of austenitic stainless steel samples were investigated, namely un-used SS321H and ex-used SS321H. The ex-used samples are belongs to a Stripper Reboiler furnace / Naphtha Hydrotreater Unit, these samples are made according to ASTM standard A-312TP321H. Their compositions with the limitations for the elements percentage according to the standard are given in table 1.

2.2. Mechanical Tests

Macro-hardness according to Vickers scale were done using a device (Nemesis 9000) with a load of 10 Kgf, the tests are supported by the relevant standard ISO-6507-1[17]. Groups of five hardness measurements were taken at room temperature for each sample (un-used material and ex-used material). Samples of these tubes were cut and prepared according to the requirement of adopted standard. Any individual of hardness measurement made in the lab will lack perfect precision that often leads to take multiple measurements. So, no one of these measurements is likely to be more precise than any other, this group of values will cluster about the true value to be measured. This distribution of data values is often represented by showing a single data point, representing the mean value of the data, and error bars to represent the overall distribution of the data. The standard error for hardness measurement was calculated (2.5008, 0.8792) for samples of the materials un-used and ex-used, respectively.

Standard charpy v-notch impact specimens were prepared according to the American Standard ASTM E23[6]. Due to tube thickness restriction, specimens with sub size dimensions were prepared, as shown in figure 1. All impact specimens were tested using a universal impact test machine (Brooks – Model IT3U). The impact tests for specimens of each type of material were performed in two different temperatures, at room temperature and at furnace service temperature (maximum wall temperature) 466°C.

Table 1,
Composition of material tubes samples.

Materials	C	Si	Mn	Ni	Cr	Ti	P	S	Fe
A-312TP321H (standard)[16]	0.04-0.1	1.0 ^A	2.0 ^A	9-12	17-19	H	0.045 ^A	0.03 ^A	Rem.
Un-Used Tube Samples	0.04	0.49	0.61	9.24	15.87	0.29	0.033	0.004	Rem.
Ex-Used Tube Samples	0.061	0.3	1.66	11.34	15.94	0.43	0.016	0.016	Rem.

A:Maximum, H:The Titanium content shall be not less than four times the carbon content and not more than 0.60%.

Table 2,
Mechanical properties for austenitic stainless steel 321H at different test temperatures.

Temperature (°C)	25		300		500		700	
Specimen Type	Un Used	Ex Used	Un Used	Ex Used	Un Used	Ex Used	Un Used	Ex Used
Yield Strength _{0.2%} (MPa)	190	219	135	191	109	182	93	153
Tensile Strength (MPa)	606	584	401	417	381	376	237	242
Elongation _{32%} (%)	69	49	40	32	37	28	56	55.6

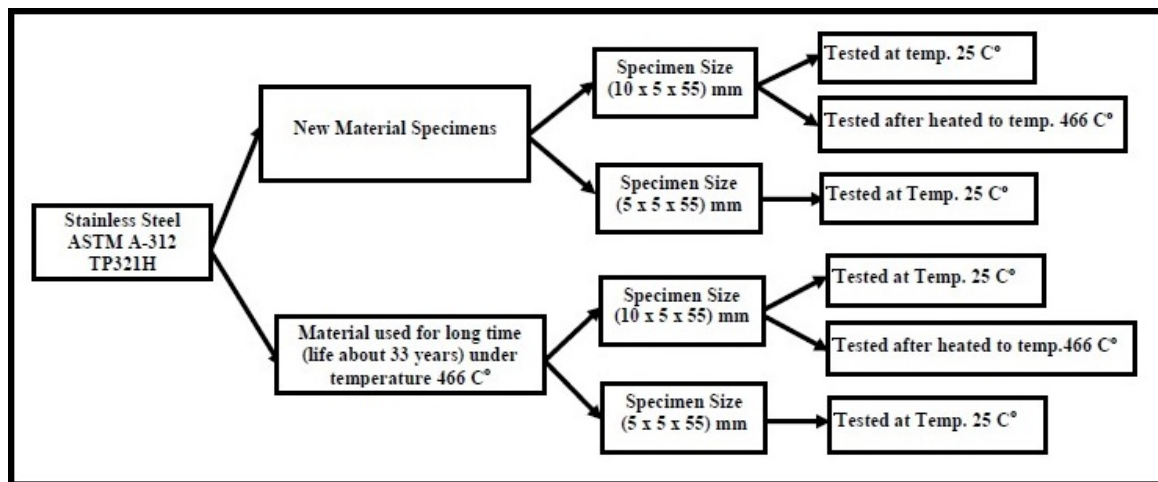


Fig. 1. Specimen's classification for Impact test.

Changes in strength between the un-used material and the ex-used one at a temperature range (from room temperature up to 700°C) can be found by performing a uniaxial tensile test using tensile machine "Shimadzu AG-25TC". Flat dog bone type specimens with thickness 3mm for this purpose were prepared according to the standard, see figure 2. Different test machine speeds were used to distinguish between the elastic and plastic zones, also these values are different either at room temperature tensile tests or for hot tensile tests. The values of yield strength (taken at 0.2% engineering strain), tensile strength, and elongation were recorded for each test temperature for both un-used material and ex-used material of austenitic stainless steel 321H, see table 2.

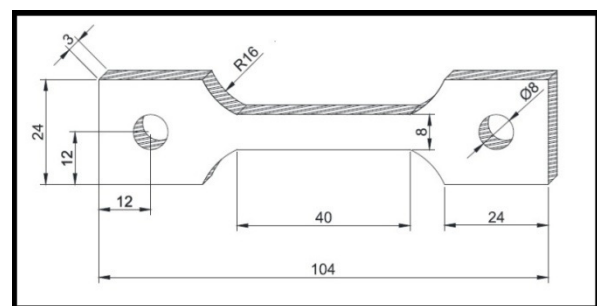


Fig. 2. Dimensions of Flat Specimen for the Uniaxial Tensile Test.

3. Results and Discussion

3.1. Hardness Behavior

As known, the easy way to sense that there is a metallurgical change happened in the metals due to temperature exposure is the hardness

measurement, but the behavior of hardness depends on steel alloy, and its carbide type precipitated. As example, the hardness of material A-213T91 decreases during creep at elevated temperature [3]. For austenitic stainless steel type 321H as seen in figure 3, the hardness increased about (11.27%) compared to the un-used material, that raising happen during the material service under operating temperature about 466°C for this long time. It is thought that this increase is related the fine titanium carbide TiC precipitated in grains at the temperature range of furnace operation, where the fine TiC precipitation increases the hardness of austenitic stainless steel type 321 up to temperature 850°C, while the coarsening of TiC precipitation softens this type of stainless steel more than 850°C [9].

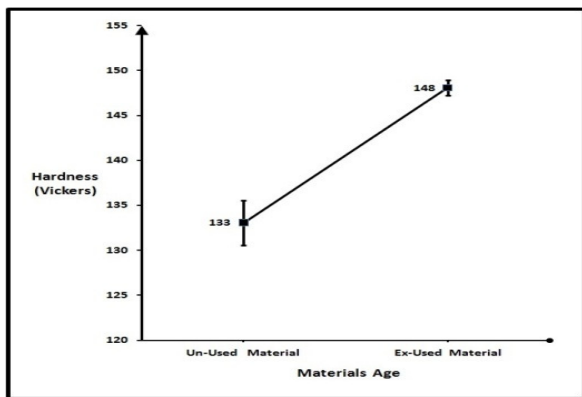
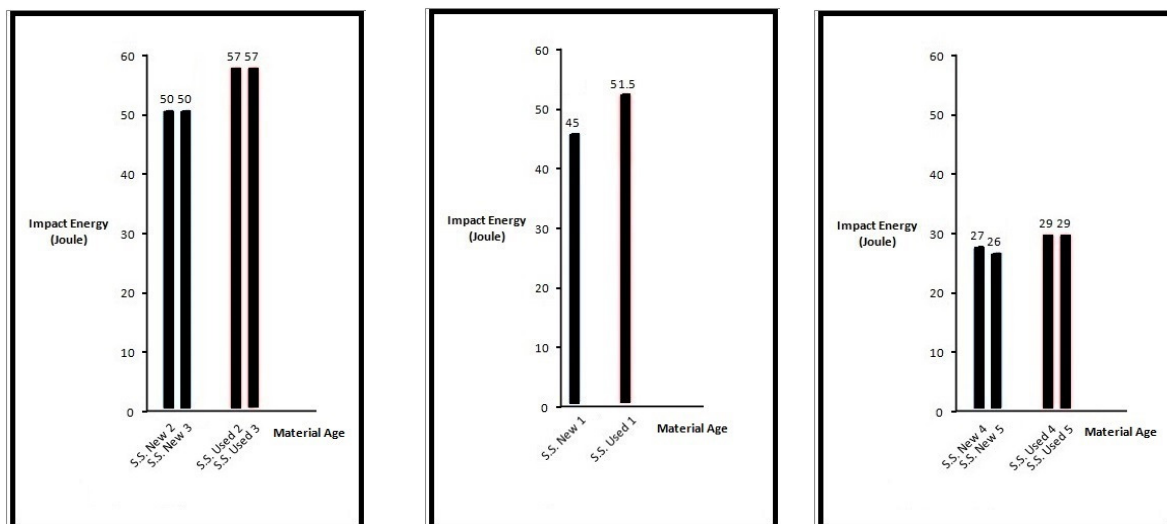


Fig. 3. Comparison between hardness of un-used and ex-used tubes of stainless steel material A-312TP321H.

3.2. Impact Energy

A comparison between the results of impact test at room temperature and at 466°C for specimen dimension (10x5x55 mm) can be seen in figures 4 (a) and 4(b) for both un-used and ex-used stainless steel 321H material. The impact energy value is directly related to the cross-sectional area of the specimens, and to make a double check for the accuracy of the measurements, another sub-size dimension (5x5x55 mm) for the specimens with 50% reduction in specimen cross section was used, see Figure 4 (c).



a. Impact test at room temperature for specimens with dimensions 10x5x55 mm

b. Impact test at temperature 466 °C for specimens with dimensions 10x5x55 mm

c. Impact test at room temperature for specimens with dimensions 5x5x55 mm

Fig. 4 Impact energy comparison between un-used and ex-used stainless steel tube samples (SA-312TP321H)

When stainless steel type 321H is aged for long time under the effect of elevated temperature exposure accompanied with creep effect in general, it shows a rising in bearing energy before break can happen, this can be measured either under room temperature impact test or at elevated temperature test, see figures 4(a) and 4(b), also the reduced specimen size (5x5x55 mm) depicts similar behavior as seen in figure 4(c). This energy increased by a percentage 14% in figures 4(a) and 4(b), as well by about 10% in figure 4(c), this is most likely due to the thermal effect occurred during the material service at the furnace operation temperature.

The temperature effect on the amount of the impact energy was also examined for the two types of stainless steel type 321H, the un-used and ex-used material, as shown in figure 5. This figure shows decreases by a percentage about 10% for each type of this material, this is most probably ascribed to the precipitation hardening effect by chromium carbides formation within the structure. This means that this decrease is due to thermal influence only, indicating that operating at this temperature for long period of time leads to increase this absorbed energy.

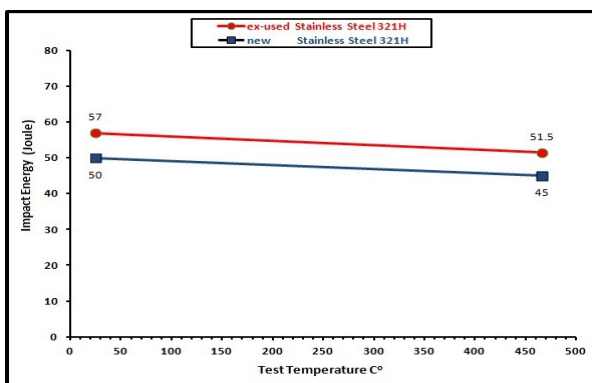


Fig. 5. Relation between impact energy and test temperature for austenitic stainless steel 321H.

3.3. Tensile Properties

The results of uniaxial tensile tests for austenitic stainless steel type 321H under different test temperatures (25°C, 300°C, 500°C, 700°C) were drawn on the same stress-strain diagram for comparison, the resulted diagram for the un-used material can be seen in figure 6, while figure 7 shows the resulted diagram for the ex-used material. The plastic deformation seems to fade with increased temperature, the maximum stresses decrease with increased temperature for the two types of samples as expected.

Tensile properties of SS321H, such as 0.2% offset yield and tensile strength, generally decrease as temperature increases, as seen in figure 8. The trend of this decreasing in properties for un-used material is the same for the ex-used material. This reduction is related to the formation of carbides at grain boundaries.

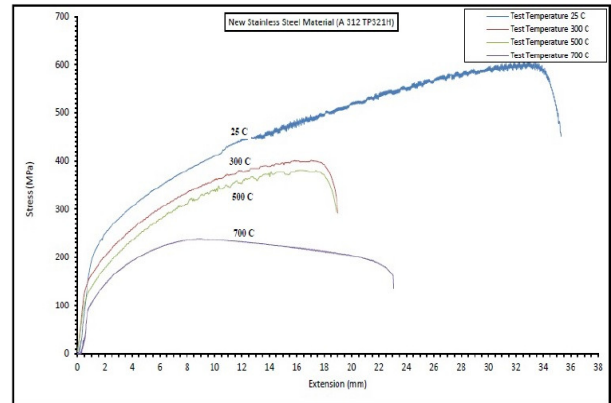


Fig. 6. Stress-strain curve under different temperatures for un-used austenitic stainless steel 321H.

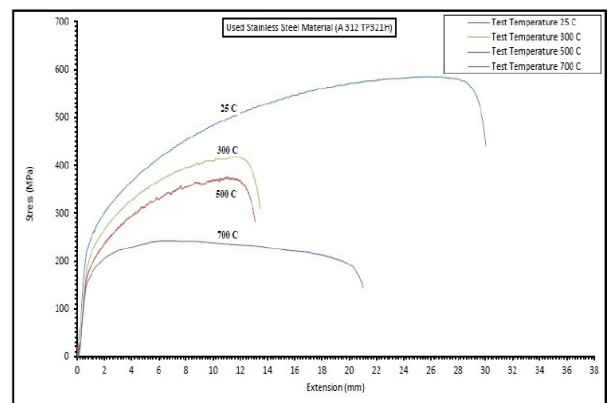


Fig. 7. Stress-strain curve under different temperatures for ex-used stainless steel 321H.

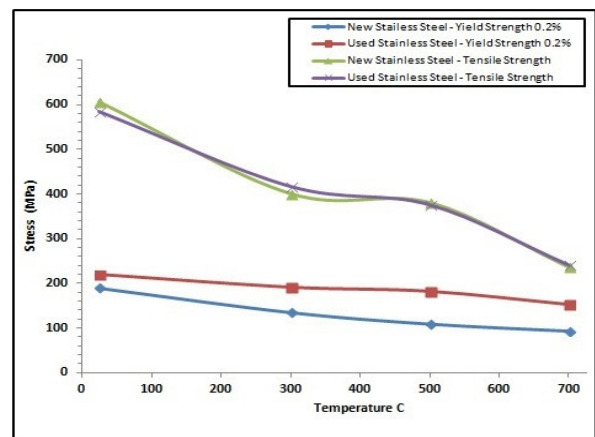


Fig. 8. Yield and tensile strength versus temperature for austenitic stainless steel 321H.

Comparisons were made between the results of uniaxial tensile tests represented by stress-strain curves as shown in figures (9 to 12) for different test temperatures, each diagram compares the tensile properties of stainless steel 321H at specific temperature between the un-used and ex-used samples. In general, stainless steel 321H lost its ability to elongate, which represents a reflection to the deterioration happened in this material due to the exposure to elevated temperature (466°C) for long time of service. On the contrary, the yield strength 0.2% offset of stainless steel 321H enhanced at each test temperature as seen in table 2, while the curves for each test temperature are in the same trend between the ex-used and un-used stainless steel 321H, but the curves for the ex-used samples are higher than the un-used ones.

The ultimate tensile stress of stainless steel 321H is changed after its long service life in this case study for stripper re-boiler furnace under maximum tube wall temperature about 466°C, this change is fluctuating, it is varying in a manner depends on the test temperature, where it is decreased at temperatures 25 and 500°C and increased at temperatures 300°C and 700°C, as shown in table 2 and in figures (9-12). So that, it is not accredited parameter to judge that the deterioration or damage due to high temperature exposure does or doesn't exist in the structure of the material.

The stress-strain curves in figures 6 and 7 reveal signs of dynamic strain aging effect for the SS321H material; this is due to alloy content, test temperature, and past service life. The common feature of dynamic strain aging is that it increases the ultimate tensile strength (UTS) and causes strengthening in a specific temperature range which depends on the strain rate as shown in table 2 for this material which is aged for long life time; this is repeated for the uniaxial tensile tests at 500°C and at 700°C, figures 10 and 12. The level of strengthening depends on the aging time and on the aging temperature.

A typical characteristic of dynamic strain aging is the formation of serrated yielding. This is often called the Portevin- LeChatelier (PLC) effect. Different serration types of stress-strain curves for the austenitic stainless steel 321H are marked on the curves of figures (9-11). The most common serration types were A, B, and D appeared on the curves up to test temperature 500°C, while at the test temperature 700°C, they didn't appear, because the material at this temperature is near the recovery and recrystallization temperatures which will decrease

the dynamic strain aging due to the extermination of dislocations.

Type A is considered as locking serrations, it is abrupt rise and then drop to a stress level below the general level. Type B has an irregular movement and is characterize by small oscillations about the general level of the curve. Type D is characterized by plateaus on the curve, it can also appear mixed with the type B [18].

Changes in mechanical properties of material as a result of service in different conditions from load, temperature, environment, etc., can be provided easily if we adopt usual examination techniques such uniaxial tensile tests to have a data or figures aid the estimation of the current internal situation of these materials, or the degree of deterioration may exist.

4. Conclusions

The following results of this work can be used for the tubes of furnaces operated at high temperatures for long time as a supportive technique to give a good indication about the remaining life estimation in short time, which could be used as a routine inspection activity during the period of scheduled shutdowns, especially when the operation life is near or overcome the design life.

- (i) Changes in mechanical properties of material as a result of service in different conditions from load, temperature, environment, etc., can be provided easily if we adopt available examination techniques to have a data or figures assist to estimate the current internal situation of these materials, or the degree of deterioration may exist.
- (ii) Hardness of austenitic stainless steel type 321H increased about (11.28%) during service for long time under temperature about 466°C, this increase is related to chromium carbide Cr_{23}C_6 and the fine titanium carbide TiC precipitated in grains at the temperature range of service.
- (iii) The absorbed energy due to impact by austenitic stainless steel 321H increased about (14%) when the material is aged for long time under the effect of elevated temperature exposure accompanied with creep effect, this is most likely due to thermal effect happened during material service at the furnace operation temperature. Also, this behavior can be recorded by the impact test with different specimens' dimensions.
- (iv) The absorbed energy due to impact by austenitic stainless steel 321H decreased about (10%) when the temperature increased up to 466°C, this behavior can be recorded if the sample

for impact test is taken from un-used material or ex-used material; this is most probably ascribed to the precipitation hardening effect by chromium carbides formation within the structure.

(v) The trend of stress-strain curve of austenitic stainless steel 321H will not change due to the exposure to elevated temperature (466°C) for long time of service and/or creep effect takes place, but the curves will be higher. The yield strength 0.2%

offset increased under these conditions, but the ability of material to be elongated under the action of axial load will decrease. The ultimate tensile stress is fluctuating in these conditions and depends on the service temperature.

(vi) Tensile properties of austenitic stainless steel 321H decrease as the temperature increases, the trend of this decreasing in properties for un-used material is the same for the ex-used material.

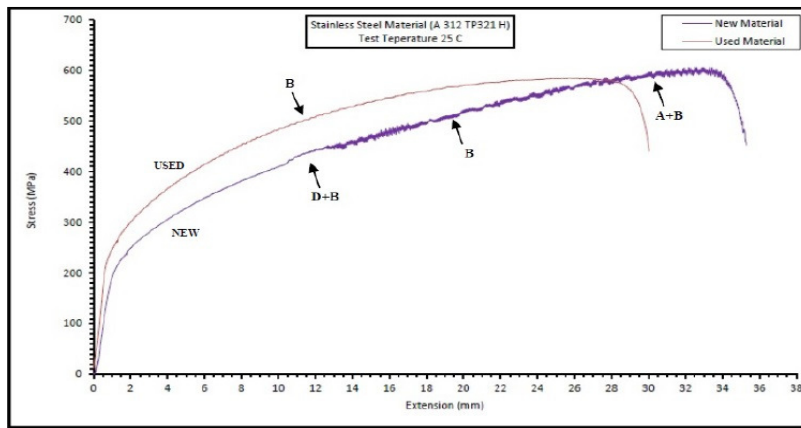


Fig. 9 Stress-strain curve at room temperature for un-used and ex-used stainless steel 321H.

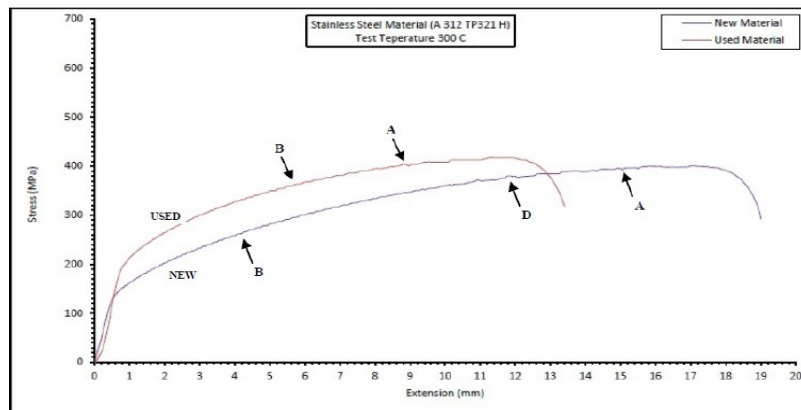


Fig. 10. Stress-strain curve at 300°C temperature for un-used and ex-used stainless steel 321H.

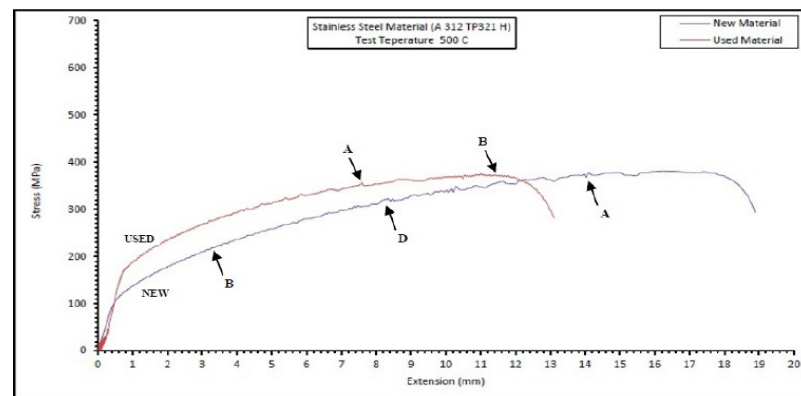


Fig. 11 Stress-strain curve at 500°C temperature for un-used and ex-used stainless steel 321H.

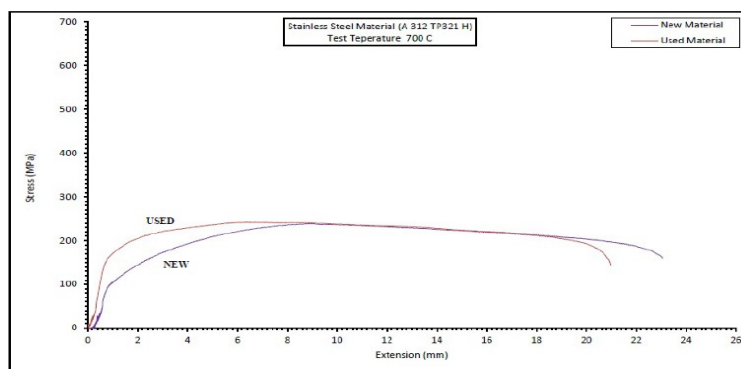


Fig. 12 Stress-strain curve at 700°C temperature for un-used and ex-used stainless steel 321H.

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تأثير العمر التشغيلي لفترات زمنية طويلة على الخصائص لسبيكة الفولاذ المقاوم للصدأ الاوستنايتي النوع 321H والتي تعمل بدرجات حرارة عالية

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الخلاصة

التغيرات الحاصلة في الخصائص الميكانيكية للمواد نتيجة للظروف المختلفة في أثناء خدمتها يمكن الحصول عليها عن طريق الاختبارات الميكانيكية، بحيث يمكن الاستعانة بالتغيرات الحاصلة للخصائص في تقدير حالة هذه المواد ودرجة الضرر المتولدة فيها وكمثال للجانب التطبيقية أنابيب الأفران الحرارية التي تعمل بدرجات حرارية عالية ولمدة عمرية تتجاوز العمر التصميمي لها. ولندرة البحوث التي تستخدم سبيكة الفولاذ المقاوم للصدأ الاوستنايتي 321H في دراستها فقد تم دراسة هذه الخصائص مثل متانة الخضوع، متانة الشد، الاستطالة، الصلادة، والطاقة الممتصة أثناء الصدمة بالاعتماد على نتائج الاختبارات العملية. فقد تم تقطيع نماذج من هذه السبيكة مستخدمة في صناعة أنابيب فرن حراري عائد لوحدة هدرجة النفط في مصفى الدورة للحصول على عينات اختبارية، فضلا عن دراسة نماذج جديدة من نوع السبيكة نفسها لأغراض المقارنة بين النتائج. بزيادة درجة حرارة اختبار الشد لحالاتي النماذج الجديدة والمستخدم سابقا اظهرت نقصان في متانة الشد، وقد اظهرت عينات حالتها المعدن المستخدم وبدرجة الحرارة نفسها الاختبار تصرف متمائل لمنحني الاجهاد-الانفعال مع زيادة في متانة الخضوع لحالة المعدن المستخدم سابقة، مع نقصان في القدرة على الاستطالة نتيجة التحميل في حالة العينات ذات العمر التشغيلي الطويل عنها في العينات الجديدة، وحصول زيادة في كل من الصلادة والطاقة الممتصة من قبل المعدن قبل حدوث الكسر بمقدار (11,28%) و (14%) على التوالي لحالة العينات ذات العمر التشغيلي الطويل مقارنة مع العينات الجديدة.