

## INTERRELATION AMONG MICROSTRUCTURE, DEVELOPMENT OF VOIDS AND THE FRACTURE BEHAVIOR OF THE NICKEL-IRON-CHROMIUM ALLOY

دراسة تأثير العلاقة بين التركيب الإنشائي ونمو التشروخ الدقيقة بين الخيانت المعدنية على سلوك الكسر في سائك الصلب مع النيكل والكروم

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الملخص

نظرا لأهمية دراسة سلوك الكسر في الأجزاء المعدنية لمكونات نظم الإنتاج الميكانيكية وعلى سبيل المثال خطوط إنتاج الأمونيا في مصانع الأسمدة (أنبوب التجميع في خط إنتاج الأمونيا) لتجنب الأضرار الفجائية لهذه الأجزاء كما حدث في واقع دراستنا الحالية فإنه من الضروري دراسة العلاقة بين التركيب الإنشائي للسائك المعدنية المصنع منها هذه الأجزاء وعلاقتها بسر التشروخ الدقيقة وتأثير ذلك على سلوك الانهيار في هذه الأجزاء الميكانيكية أثناء عمليات وظروف التشغيل المعتادة. وقد تركز البحث على استخدام نظريات ومعايير ميكانيكا الكسور لتقدير مدى تأثير معامل الإطالة ومقدار المطوية المعدنية باختلاف في التركيب الإنشائي للسبيكة وتأثير ذلك على مقاومة السبيكة المعدنية لنمو التشروخ الدقيقة أثناء عمليات التشغيل. وقد خلصت الدراسة إلى عدم موافقة التركيب الإنشائي والكيميائي للعناصر الأساسية والعناصر الإضافية لمكونات للسبيكة المصنع مسبقا أنبوب التجميع مما أدى إلى ترسيب بعض العناصر الإضافية الخفيفة بين الخيانت المعدنية أدى إلى نشأة نواة للتشروخ الدقيقة مع عدم تحاسن في التركيب الإنشائي للخيانت المعدنية مما أثر على مطوية السبيكة ومقدار معامل الإطالة الذي يعبر عن مقدار مقاومة السبيكة لنمو التشروخ الميكانيكية وسلوك الكسر تحت ظروف التشغيل المختلفة.

### ABSTRACT

In order to understand the fracture behavior of the fractured tube of the ammonium pipeline in the fertilizer production system, it is necessary to take into consideration the interrelation among microstructure, development of voids and the mechanical behavior of the material from which the tube is made.

A study of the influence of microstructural parameters on fracture toughness of the tube material (Nickel-Iron-Chromium alloy) has been made. Using the  $J$  integral concept,  $J$ -resistance curves have been obtained and the influence of strain hardening on fracture toughness has been evaluated. Hence, the tearing instability concept was applied to investigate the fracture process. The relationship between critical stress intensity factor and the microstructural parameters has been taken into account. Ductile fracture occurring by the formation, growth and coalescence of voids around the inclusions was described. In addition, micro-fractographic examination has been carried out to establish a possible correlation between the mechanical properties and the micro-mechanisms of fracture.

### KEYWORDS

Microstructural characteristic; Material chemical composition; Mechanical and Microstructural characteristic; Tearing modulus;  $J$ -integral resistance; Tearing mode stress intensity factor; Crack growth; Microstructure parameters; Non metallic inclusions; Strain-hardening coefficient; Fracture toughness parameter.

### 1-INTRODUCTION

Cracks will initiate and grow at some local weaknesses in the material, due to presence of voids, micro-cracks, flaws, and the like [1-7]. Fatigue crack propagation is influenced by numerous factors, including the nature, type and magnitude of loading, length and shape of crack, size and shape of component, environment, material and microstructure [8-10]. Often the manufacturing process may be responsible for providing the source of crack nucleation. For example, in a welded structure, the weld will contain small defects, which may be sufficiently damaging to start the crack growing from the first few stress cycles, possibly from more than a

single nucleation source. In assessing the fatigue integrity of a structure or structure component, due account must be taken of the residual stress caused by expansion and contraction during the manufacturing processing such as casting, forging and welding processes [10-15].

## 2-ANALYSIS

Consider the brittle failure of the fractured tube (collector) of the ammonium pipeline in the fertilizer production system, which is a valuable illustration of the problem of assessing the significance of real defects. In this regard, it is important to note that, investigation of the fractured tube indicate that, the failure was due to tearing mode of fracture.

It seems clear that the deformation capacity of any structural member is closely related to some particular parameter such as stress-strain properties and microstructure parameters of the material used. Consequently, a detailed investigation of these mechanical and microstructural parameters and their effect on the fracture behavior of the structure is necessary.

### 2-1 Tearing Instability Concept Analysis

The concept of tearing modulus,  $T$ , has been developed based on the  $J$ -integral resistance curve and the two non-dimensional quantities,  $T_{mat}$  and  $T_{app}$  [15-19]. The value of  $T_{mat}$  represents all essential properties of the material, while  $T_{app}$  refers to the geometric configuration of the specimen. Both quantities may be defined as follows:

$$T_{mat} = \left[ \left( \frac{E}{\sigma_f^2} \right) \frac{dJ_{mat}}{da} \right] \quad (1)$$

$$T_{app} = \left[ \frac{4L}{D^2} (2a) \right]$$

The deformation condition of stability of crack growth is given by the following inequalities:

$$T_{mat} > T_{app} \quad \text{stable condition} \quad (2)$$

$$T_{mat} < T_{app} \quad \text{unstable condition}$$

In these expressions: "E" is modulus of elasticity " $\sigma_f$ " true fracture stress. "a" is the relevant crack size.  $J_{mat}$  is the value of  $J$ -integral following the material resistance curve. L is the effective specimen length. D and 2a are the gauge diameter and the crack length of the specimen respectively.

Although this implies that only small amounts of crack growth are allowed, the  $J$ -integral tearing modulus approach has been shown successful in predicting stable crack growth and ductile fracture instability. The linear elastic  $J$ -integral is given by the following:

$$J = K_{III}^2 / E_p \quad (3)$$

Where  $K_{III}$  is the tearing mode stress intensity factor,  $E_p = E/(1-\mu^2)$  for plane strain and E for plane stress. E is the modulus of elasticity, and  $\mu$  is Poisson's ratio.

A closed form expression for tearing mode stress intensity factor, is given in a modified form as follows:

$$K_{III} = 0,25 p_i d_i^{1/2} \sqrt{\pi \beta (2a/t)} \left\{ \sqrt{\left( \frac{t}{d_i} \right)} \left[ 1 + \left( \frac{t}{d_i} \right) \right] \right\}^{-1} \quad (4)$$

Where  $\beta$  is curvature parameter,  $t$  is the tube thickness,  $d$ , is the tube internal diameter and  $p$ , is internal pressure.

$$\beta^2 = \left( \frac{t}{d} \right) \sqrt{12(1 - \mu^2)}$$

The applied tearing modulus,  $T$ , is obtained from Equation (1) by differentiating the  $J$ -integral of Equation. (3), with respect to crack depth,  $2a$ , and then using equation (4) to obtain  $T$ .

#### 2-1-1 Application of the tearing modulus concept

Tensile tests were performed. The specimens were manufactured from the trunk of a supplied spare part tube and from the recommended material (Nickel-Iron-Chromium alloy).

Figure 1. Shows the tensile stress-strain relationship for the recommended material and the supplied tube material. The figure indicates the vast difference in the deformation capacity between these two materials.

The mechanical properties resulted from the tensile tests for the recommended material as well as the supplied tube material (Nickel-Iron-Chromium alloy) are reported as given in tables 1a and 1b.

TABLE 1a. Mechanical Properties experimentally determined for the recommended material (Nickel-Iron-Chromium alloy).

Alloy	Condition	Tensile Strength MPa	Yield Strength (0.2% Offset) MPa	Elongation %
Nickel-Iron-Chromium	Hot-finished annealed, or cold drawn annealed	542	266	34

TABLE 1b. Mechanical Properties experimentally determined for the supplied tube material (Nickel-Iron-Chromium alloy).

Alloy	Condition	Tensile Strength MPa	Yield Strength (0.2% Offset) MPa	Elongation %
Nickel-Iron-Chromium	Non-heat-treated material	455	318	9

In order to further understanding of the fracture behavior of the recommended material as well as the fractured tube material, the tearing instability criterion was applied as follows (see Appendix-A):

#### The first step is:

Using the appropriate data experimentally determined for the recommended material (Nickel-Iron-Chromium alloy) that recommended according to ASTM standards and henceforward comparing the obtained values of  $T_{mat}$  and  $T_{app}$  as follows:

$T_{mat}, T_{app}$  it is seen that

$$T_{mat} > T_{app}$$

Consequently, according to Eq. (2) the condition of the recommended material is stable

#### The second step is:

Using specimens cut-off and machined from the supplied tube material, static monotonic tests were performed and the respective values of  $T_{mat}, T_{app}$  recorded, resulting in:

$$T_{mat} < T_{app}$$

This inequality corresponds with the process of unstable deformation. Thus, the properties of the supplied tube material effect the deformation capacity and the mechanical behavior of the collector tube. Thereupon, the material from which the tube was made is able to develop tearing instability that leads to crack propagation under the operating conditions.

A parametric study for the applied tearing modulus has performed and the results are shown in Fig. 2 and 3.

## 2-2. Influence of the Microstructural Parameter on the Critical Stress Intensity Factor

The relationship between the critical stress intensity factor, tensile properties and the microstructure parameter of materials has been investigated [20-24]. It is now generally accepted that a correlation between tensile properties and fracture toughness parameter that could be considered as the critical stress intensity factor value exists upon Mode-III loading. However, the critical stress intensity factor  $K_{Icritical}$  may be expressed as follows:

$$K_{Icritical} = \left[ \frac{S' \cdot \pi (l+n)}{(l-\mu^2)} \cdot \sigma_y^{(l-n)} \cdot \epsilon_f^{(l+n)} \cdot E^{(l+n)} \right]^{1/2} \quad (5)$$

This formula shows that  $K_{Icritical}$  is linked to four tensile quantities  $\sigma_y$ ,  $E$ ,  $\epsilon_f$ ,  $n$  and to microstructure parameter  $\delta$ . Thus,  $K_{Icritical}$  can be calculated after measuring  $\delta$  by metallographic observations.

Where  $\sigma_y$  is the yield strength,  $E$  is the modulus of elasticity,  $\epsilon_f$  is the true fracture strain in uniaxial tension,  $n$  is the strain-hardening coefficient of the material, considered equal to the uniform elongation strain at the instability of tensile specimen and  $\delta$  is the inclusion spacing.

Figure 4, shows the variation of critical stress intensity factor for the recommended and supplied tube materials as a function of microstructure parameter,  $\delta$ .

## 2-3. Heat Treatments and Microstructure Control

Many forming and joining processes involve heating and cooling the metal and thus accidental heat treatment may result; if these facts are not realized then serious consequences may occur such as non-homogeneous microstructure. Moreover developing residual stress, segregation of added elements: non-controlled grain size, increasing the brittleness of the material, developing intergranular and trans-granular cracks that propagate under normal operation conditions may be the result [24-26]. An example is the fractured tube, which is studying in this work.

Fig. 5 shows illustrated drawing for the fractured tube. Fig. 6 shows photomicrograph of the recommended material and the supplied tube material. Consideration of these photomicrographs indicates the difference in the microstructure.

## 3- INVESTIGATION ANALYSIS

Although three samples of test pieces were taken-up from the same fractured tube trunk, the results of the microscopic investigation revealed that there is vast difference between the grain microstructure of the three test samples (A, B and C). In addition, the material from which the tube was made is non-homogeneous material.

Figure 7 shows some typical formations and growth of intergranular cracks that found in the fractured tube material. These photomicrographs show intergranular and transgranular nonmetallic inclusions in austenitic matrix with very small voids were enveloping the inclusions.

The result of the chemical composition analysis for the fractured tube material as reported by CMRI is given in the following table.

Chemical composition analysis as reported by CMRI (Composition Limits, %)											
Sample	C	Si	Mn	P	S	Cr	Ni	Ti	Al	Cu	Fe
A	0.121	0.785	0.862	0.015	0.004	20.66	31.36	0.058	0.137	0.119	Rest
B	0.122	0.755	0.844	0.015	0.003	20.62	31.57	0.054	0.129	0.121	Rest
C	0.120	0.763	0.845	0.015	0.003	20.64	31.50	0.055	0.131	0.120	Rest

The mean inter-particle spacing between the non metallic inclusions (Inclusion mean spacing) = 76-90 nm, strain-hardening coefficient  $n=0.086$ .

The Nickel-Iron-Chromium alloy recommended material is chosen according to ASTM standards [ASTM 87]. The properties that characterize the recommended material were matched with the characteristic properties of

UNS N08810 that is normally employed in service temperatures above 593°C. The material grain size, nearest (0.060 mm), with the following chemical composition.

Chemical composition of the recommended material (recommended according to ASTM). "Nickel-Iron-Chromium Alloy UNS N08810" (Composition Limits, %)											
Sample	C	Si	Mn	P	S	Cr	Ni	Ti	Al	Cu	Fe
Collector tube	0.05 to 0.10	1.0 max	1.5 max	-	0.015 max	19.0 to 25.0	30.0 to 35.0	0.15 to 0.60	0.15 to 0.60	0.75 max	Rest

The mean inter-particle spacing between the non-metallic inclusions (Inclusion mean spacing) = 120-140  $\mu$ m, strain-hardening coefficient  $n=0.32$ .

#### 4-RESULTS AND DISCUSSION

The applied J-integral tearing modulus method of analysis for tubes containing intergranular cracks, voids or flaws subjected to internal pressure was employed to analyze the fracture behavior of the fractured tube. The tearing modulus analysis based upon the closed-form of stress intensity factor given by equations (4) and (5).

The J-integral and tearing modulus were applied in this investigation for predicting stability of crack growth in the tube material. Observation of the curves plotted in Figures (2) and (3) indicate that, the crack is stable as long as the calculated applied tearing modulus does not exceed the material tearing modulus. On otherwise, when the applied J values equal the material J values.

The method of analysis followed herein was performed to assess crack instability. Hence, plotting the applied tearing modulus versus normalized J for various values of crack depth to tube thickness ratio,  $a/t$ , for different values of tube thickness to tube inner diameter ratio,  $t/d$ . Thereafter superimpose the material tearing modulus curve as shown in Fig. 3. Moreover, the maximum crack size a structure member can be tolerating at the particular stress level is indicated, for Nickel-Iron-Chromium Alloy as a recommended material for tube manufacturing.

For the sake of understanding the interrelation between the material microstructure and the fracture toughness that control the fracture behavior of a structure, hence, the nucleation of ductile fracture from intergranular cracks or voids located between major metallic inclusions. A link is established between mechanical and microstructural quantities expressed as given in Equation (5).

Careful consideration of Figure 4, that plotted on the base of Equation (5) hypothesizes that crack tip blunts, which is of the same order of magnitude as spacing between major non-metallic inclusions,  $s'$ . Then the onset of crack advance intervenes in same way as in a specimen with blunt notch. Consequently, a procedure to drive  $J_{IC}$  data from the values of the applied J-integral at fracture nucleation was carried out. Blunt notch specimens with notched end radii greater than  $s'$  has been devised and applied to compare calculated and experimental  $J_{IC}$  pertaining to Nickel-Iron-Chromium Alloy that recommended in this investigation. In addition, it can be stated that the fracture toughness  $K_{IC}$  or  $J_{IC}$  in ductile ruptures is controlled both by the spacing between inclusions and by total second phase volume.

Based on the results of test investigations and the analytical-experimental study of the interrelation among microstructure and development of voids it is, therefore, necessary to identify the various factors affecting the mechanical behavior of any structure component. The effects to be taken into account is the microscopic and consequences of heating and cooling the Nickel-Iron-Chromium alloy. The most important of microscopic or metallurgical changes are those which affect the strength and, in particular, the ductility of the alloy. Consideration of Figure 1 identifies the effects of the microscopic changes on the strength and ductility of the fractured tube material and recommended material. Thus, it is the loss of ductility, which goes with increasing strength that of greatest cause for our concern.

Furthermore it has been shown that, manufacturing and joining process followed by non-heat-treated resulting in non-homogeneous grain microstructure as well as grain size, in addition, effected during operation cyclic. This being clearly shown in the microstructure photographs Figure 7.

The non-homogeneous grain microstructure and grain size lead to crack propagation under normal operational condition. In addition, the operational temperature, which may reach the recrystallization temperature of the tube alloy, may affect the low-melting-point metals. Nonetheless, iron waved with low-melting-point elements such as copper, aluminum and titanium etc. These elements weaken the grain boundaries by reducing their

surface energy and the intergranular cracks will be generated in the grain boundaries. Thus, they act in a manner similar to stress raisers, such as notches. However, the crack initiation has been related to the fact that the increase of the operating temperature does modify the microstructural parameter that play a role in controlling the fracture mechanism of micro-void nucleation and coalescence

### 5-CONCLUSION

The tearing instability concept and the tearing modulus criterion were used to investigate the tensile deformation of the Nickel-Iron-Chromium Alloy. In addition, nucleation of voids was studied to understand the interrelation among microstructure and fracture toughness, which control the fracture behavior of the structure and structure components. Accordingly, it is concluded that:

- 1- The stability of the fracture toughness has been related to the fact that, the changes in the microstructural parameters play a role in controlling the fracture mechanism of micro-void nucleation and coalescence.
- 2- The changes in grain microstructure influence voids nucleation and growth under normal operational condition.
- 3- Void nucleation at carbides occurs at low strains and these voids dominate the fracture process because of the small spacing between the carbide particles especially along the grain boundaries giving a significant reduction in toughness and ductility.

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## APPENDIX

## A-EXPERIMENTAL PROCEDURE

It is a common practice to determine the tensile properties of a material using round bar test-specimen, such as in this investigation, according to the standard BS18 (1971). Static tensile tests were performed at a loading rate of 0.5 mm/min on cylindrical specimen,  $d=12$  mm. The specimens were manufactured from the trunk of a supplied spare part tube and from the recommended material (Nickel-Iron-Chromium alloy) of thickness equal 35-mm with their axes parallel to the manufactured tube axis. After the general yielding and at the beginning of the necking the strain hardening coefficient  $n$  reached the values of 0.32 for the recommended material (Nickel-Iron-Chromium alloy). Moreover, strain-hardening coefficient  $n$  reached the value of 0.086 for the supplied tube material. On completion of the necking, the value of  $n$  increased to 0.38 for the (Nickel-Iron-Chromium and kept constant for the supplied tube material. The ultimate failure of the specimens was characterized by gross yielding in the gage length. In order to develop the present understanding of the fracture, the tearing instability criterion was applied.

The concept of tearing modulus,  $T$ , has been developed based on the  $J$ -integral resistance curve and the two non-dimensional quantities,  $T_{material}$  and  $T_{applied}$  [15-19]. The value of  $T_{mat}$  represents all essential properties of the material, while  $T_{app}$  refers to the geometric configuration of the specimen. Both quantities may be defined as follows:

$$T_{mat} = \left[ \left( \frac{E}{\sigma_f^2} \right) \frac{dJ_{mat}}{da} \right] \quad (i)$$

$$T_{app} = \left[ \frac{4L}{D^2} (2a) \right]$$

The deformation condition of stability of crack growth is given by the following inequalities:

$$\begin{aligned} T_{mat} > T_{app} & \quad \text{stable condition} \\ T_{mat} < T_{app} & \quad \text{unstable condition} \end{aligned} \quad (ii)$$

In these expressions: "E" is modulus of elasticity " $\sigma_f$ " true fracture stress, "a" is the relevant crack size,  $J_{mat}$  is the value of  $J$ -integral following the material resistance curve, L is the effective specimen length, D and 2a are the gauge diameter and the crack length of the specimen respectively.

At the beginning of the necking process no internal crack in the body of the specimen gauge length was assumed,

$$\text{i.e.} \quad 2a = 0$$

$$\text{Therefore} \quad T_{app} = 0$$

Using the appropriate experimental data for the Nickel-Iron-Chromium alloy, we obtain

$$T_{mat} = 225.1$$

The following values were applied

$$dJ/da = 306.8 \text{ MPa}$$

$$E = 2.1 \times 10^5 \text{ MPa}$$

$$\sigma_f = 535 \text{ MPa}$$



Comparing  $T_{app}$  and  $T_{mat}$  it is seen that

$$T_{mat} > T_{app}$$

Consequently, according to equation (ii) the condition is stable.

Now considering the point of failure for the supplied tube material, it will be realized that the situation is different. Three necking tests were performed and the respective values of  $T_{app}$  and  $T_{mat}$  recorded, resulting in.

$$T_{mat} < T_{app}$$

This inequality corresponds with the process of unstable deformation.

### B-Application of Through-Thickness Yielding Criterion.

By using concepts of linear-elastic fracture mechanics, a quantitative approach to the development of toughness requirements is based on the requirement that in the presence of sharp crack in an engineering structure, through-thickness yielding should occur before fracture. It has been shown that a toughness criterion for steels to obtain through-thickness yielding before fracture can be developed in terms of yield strength and structure thickness as follows [20]:

$$K_{IC} > c \sigma_y (2a_{max} < t)^{1/2} \quad (iii)$$

Accordingly, the required value of  $K_{IC}$  that could satisfy steel using requirement could be obtained according to the through-thickness-yielding criterion as follows:

$$K_{IC} = c \sigma_y (2a_{max})^{1/2}$$

Using the experimentally determined data for a particular material (Nickel-Iron-Chromium alloy), thus the maximum crack size a structure member can tolerate at the particular stress level is

$$2a_{max} = 1/c [K_{IC} \text{ or } K_{max} / \sigma_y]^2 \quad (iv)$$

$$c = 2 \pi / (1 + 3\beta^2)$$

Where  $\beta$  is the tube curvature parameter.

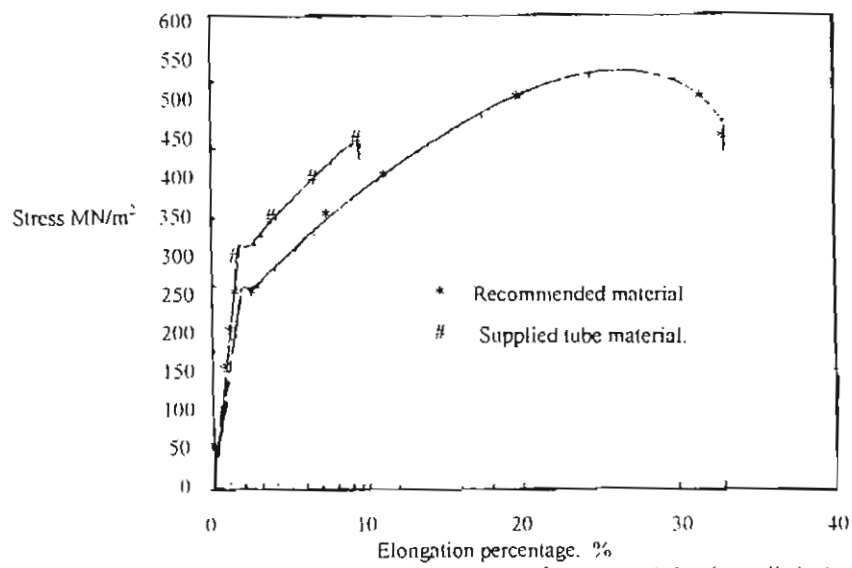


Fig. 1a. Comparison between the tensile properties of recommended and supplied tube material.

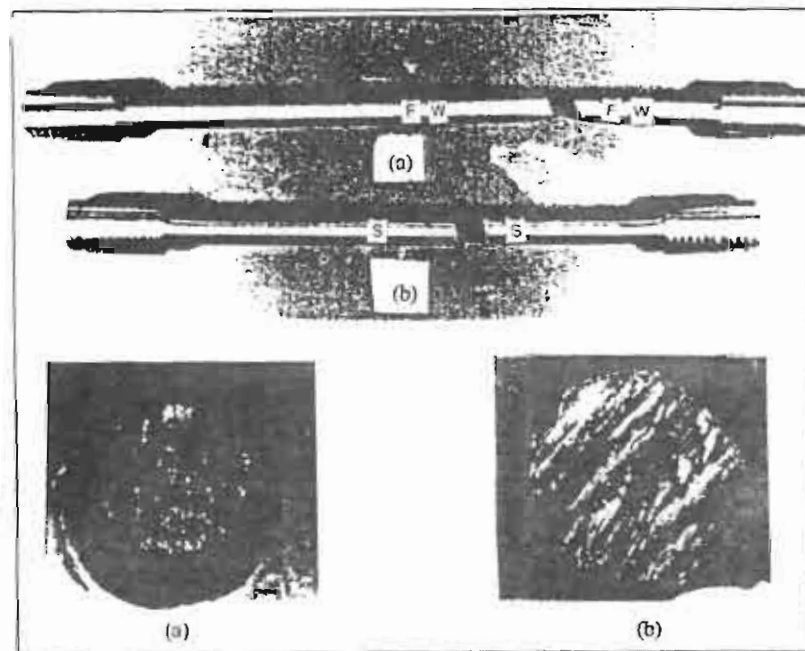


Fig. 1b. Tested specimens and fractured surface after performing the tensile test. for a) Recommended material and b) Supplied tube material.

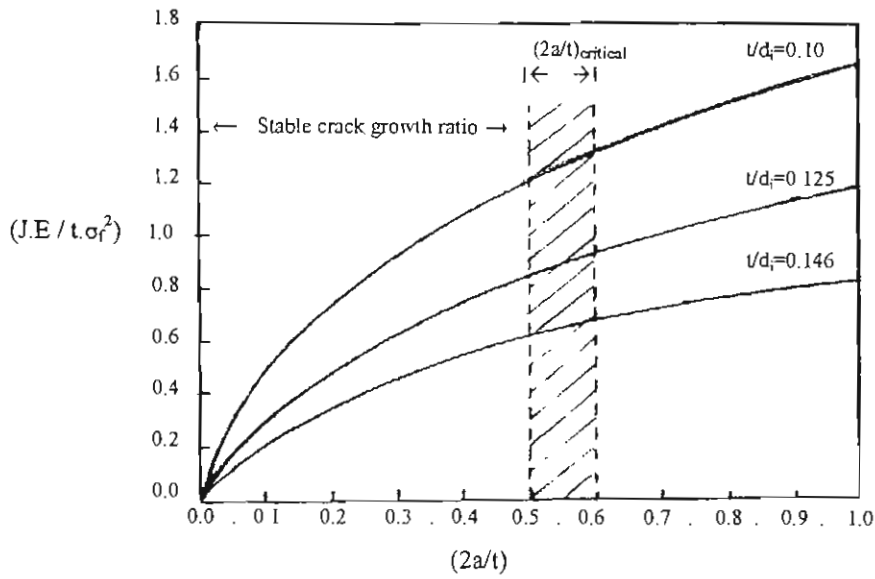


Fig. 2. J-integral vs crack depth / tube thickness for values of  $v/d_i=0.1, 0.125$  and  $0.146$ .

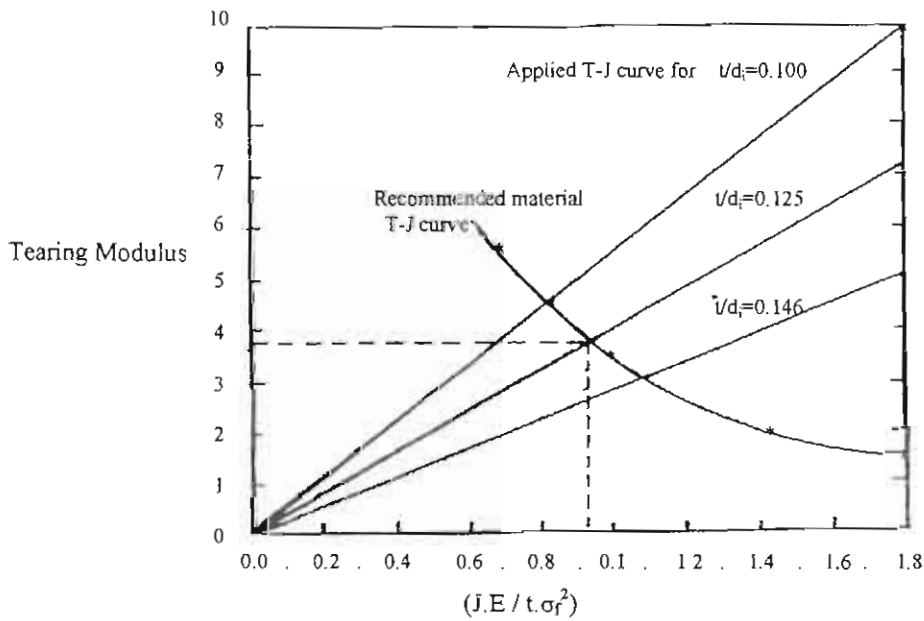


Fig. 3. Tearing modulus vs normalized J-integral for various values of  $v/d_i=0.1, 0.125$  and  $0.146$  with the T-J curve for the recommended material.

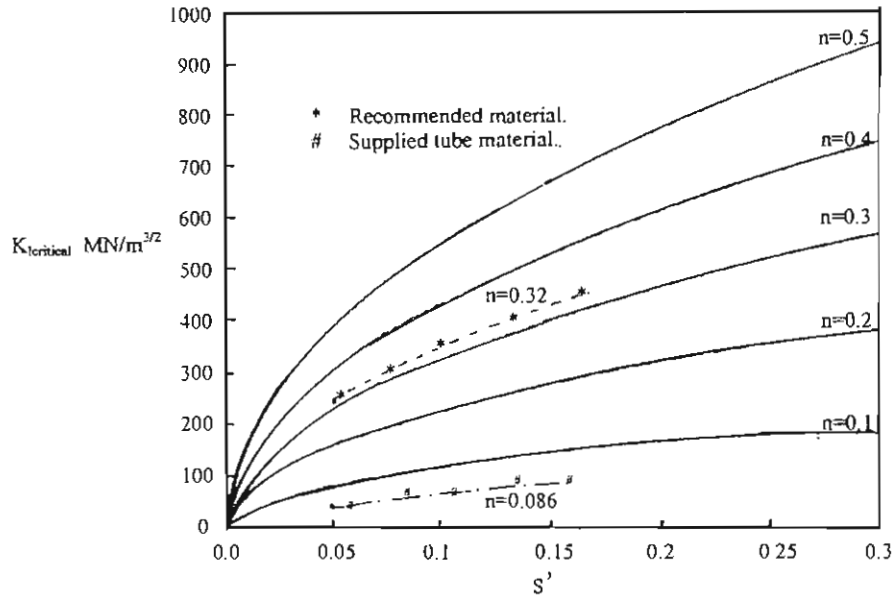


Fig. 4. Variation of critical stress intensity factors of Nickel-Iron-Chromium alloy as a function of microstructure parameter  $s'$ , at different stress hardening values  $n$ .

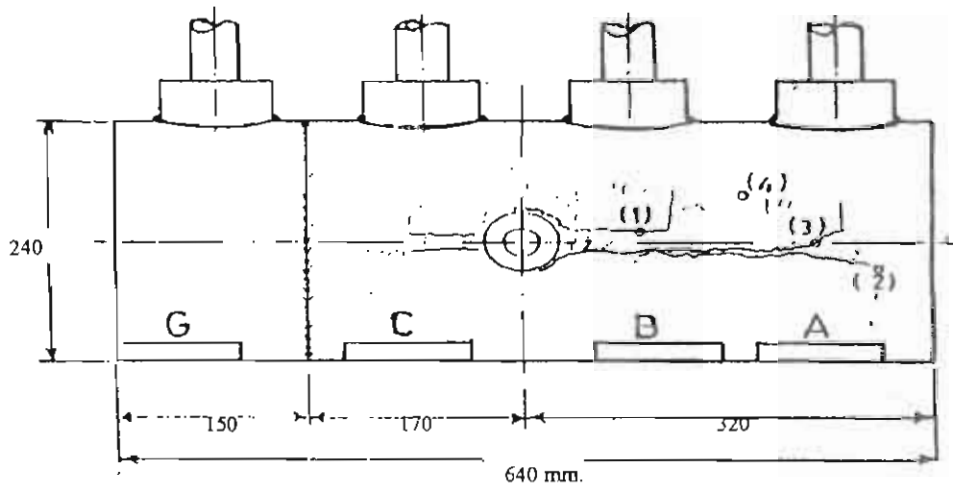
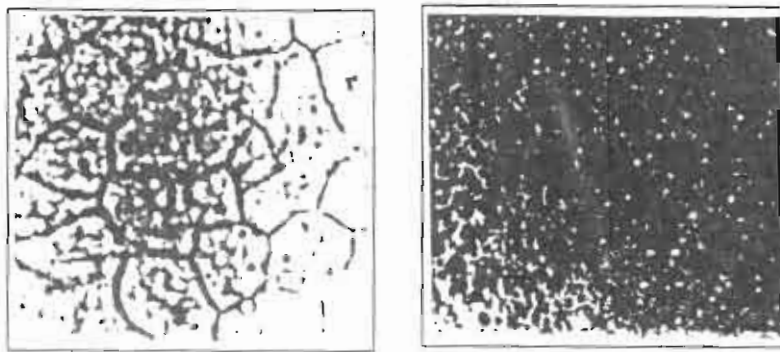


Fig. 5. Fractured tube geometry and crack branches locations.



(a) (b)  
Fig. 6. Photomicrograph of the Nickel-Iron-Chromium alloy.  
a) Recommended material and b) Supplied tube material

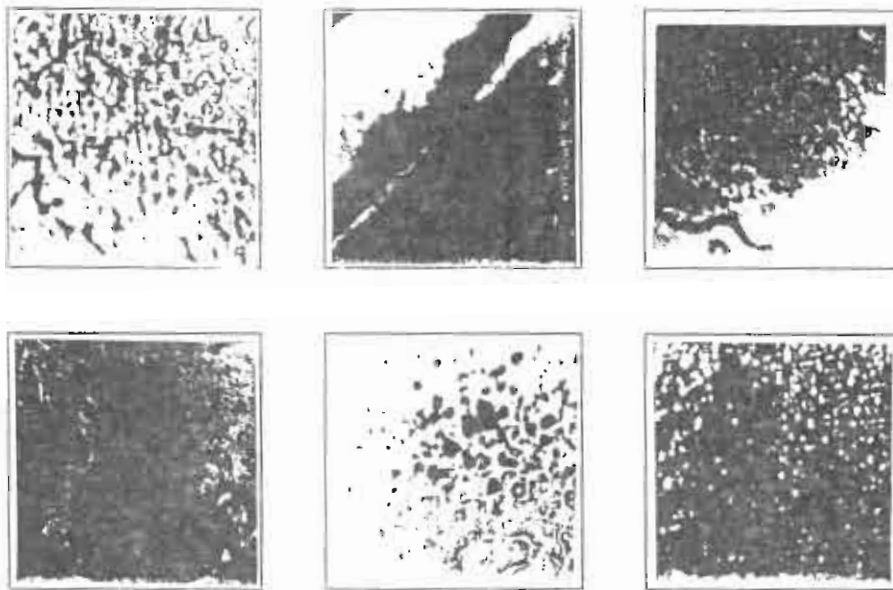


Fig. 7. The microstructure of the tested samples that taken-off from the fractured tube trunk. The microstructure consist of some intergranular and transgranular non-metallic inclusions in an austenitic matrix.