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DETERMINATION DE LA TENACITE DE COUDES DU CIRCUIT PRIMAIRE VIEILLIS EN SERVICE A L'AIDE DE MINI-EPROUVETTES CT PRELEVEES EN PEAU EXTERNE

FRACTURE TOUGHNESS ASSESSMENT OF IN-SERVICE AGED PRIMARY CIRCUIT ELBOWS USING MINI C(T) SPECIMENS TAKEN FROM OUTER SKIN

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SYNTHÈSE :

Les aciers inoxydables double couche coulés de type CF8M qui sont employés dans les coudes du circuit primaire sont soumis à une fragilisation par vieillissement thermique à leur température en service d'environ 300°C. Ce phénomène affecte leurs propriétés de résistance à la rupture. Pour évaluer la résistance à la rupture résiduelle de ces coudes, on effectue des estimations grâce à des formules prédictives reposant sur la composition chimique et sur les conditions du vieillissement, et qui fournissent des valeurs sûres. Toutefois, dans le cas des matériaux les plus sensibles, il est important d'obtenir des estimations plus précises. On a donc envisagé une nouvelle méthode de détermination, fondée sur des tests appliqués à des mini-éprouvettes CT prélevées sur l'enveloppe extérieure des coudes en service.

L'utilisation de mini-éprouvettes CT pour évaluer de la résistance à l'arrachement des aciers inoxydables coulés paraît, à première vue, difficile, notamment en raison de la structure métallurgique très grossière de ces aciers : de petites éprouvettes peuvent-elles être représentatives de plus gros volumes (principalement des éprouvettes normales IT-CT), et n'apporteront-elles pas une dispersion excessive ? Pour répondre à ce genre de questions, un programme de validation expérimentale a été entrepris : le programme terminé indique que cette méthode convient, ce qui conduit à la proposition d'orientations visant à optimiser l'analyse des résultats expérimentaux.

La méthode est ensuite appliquée à un coude en service. Les résultats obtenus montrent une bonne corrélation avec les estimations d'épaisseur fournies par nos formules prédictives. Ceci contribue à son tour à la validation de la méthodologie générale utilisée en France pour la justification des coudes de circuit primaire.

EXECUTIVE SUMMARY :

Type CF8M cast duplex stainless steels used in the primary circuit elbows of pressurized water reactors are subject to thermal aging embrittlement at their service temperature, around 300°C. This phenomenon affects their fracture toughness properties. In order to assess the residual fracture toughness of these elbows, estimations are made through predictive formulae based on chemical composition and aging conditions, which provide safe values. However, in the case of the most sensitive materials, it is important to obtain more accurate estimations. A new method of determination was thus considered, based on the testing of mini-CT specimens taken from the skin of in-service elbows.

The feasibility of using mini-CT specimens to evaluate the tearing resistance of cast duplex stainless steels seems at first sight difficult, in particular because of the very coarse metallurgical structure of these steels: will small specimens be representative of larger volumes (mainly regular IT-CT specimens) and will they not induce too much scatter? In order to answer such questions, an experimental validation program has been undertaken: the completed program shows that the method is relevant and leads to proposed guidelines which aim at optimizing the experimental results analysis.

Then the method is applied to an in-service elbow: the results obtained are found to be in good agreement with the toughness estimations given by our predictive formulae. This subsequently contributes to the validation of the general methodology used for the justification of French primary circuit elbows.

FRACTURE TOUGHNESS ASSESSMENT OF IN-SERVICE AGED PRIMARY CIRCUIT ELBOWS USING MINI C(T) SPECIMENS TAKEN FROM OUTER SKIN

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I INTRODUCTION

Type CF8M cast duplex stainless steels used in the primary circuit elbows of pressurized water reactors are known to undergo thermal aging embrittlement at their service temperature, around 300°C. This phenomenon affects their mechanical properties, mainly their fracture toughness (tearing resistance) [1, 2], which needs to be evaluated for mechanical analyses.

Fracture toughness estimation methods through predictive formulae using chemical composition and aging conditions are available (e.g. [3]), which provide safe values. However, in the case of the most embrittled elbows, more accurate evaluations are also needed. A new method was therefore considered : it consists of taking relatively thin slices of material from elbow surfaces and performing tearing resistance tests on very small fracture mechanics specimens (0,4T-CT with thickness reduced to 5 mm), called mini-CT.

Given the coarse macrostructure of the cast duplex stainless steels and the level of scatter usually encountered on tearing resistance curves using 1T-CT specimens, the use of mini-CT specimens is not obviously relevant. A validation program was thus undertaken before starting taking samples from in-service elbows.

This paper deals with the development and application of mini-CT specimens testing. It is divided into three parts : the first part presents experimental procedures, the second shows the results of the validation program and the third is devoted to the application to an in-service elbow.

II EXPERIMENTAL PROCEDURES

All tearing resistance tests were carried out according to the GFR procedure [4]. Except few differences, this procedure is the French equivalent to the ASTM E813-89 standard.

Specimen preparation

Two types of specimens are used in this paper : 1T-CT

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TABLE 1 : PRESENTATION OF THE MATERIALS STUDIED IN THE VALIDATION PROGRAM

Type of product	Mark	Thermal aging treatment	KCU at 20°C* (daJ/cm^2)	KCV at 320°C** (daJ/cm^2)
90° elbow	EL	3000h at 400°C	1,0	1,5
Plate	CC	1000h at 400°C	1,0	2,8
Plate	DI	10000h at 400°C	1,8	3,2
50° elbow	EK	30000h at 350°C	1,4	3,8
Plate	DI	700h at 400°C	2,8	8,0
50° elbow	EK	30000h at 325°C	2,3	10,4

* KCU : Charpy U-notch impact strength
** KCV : Charpy V-notch impact strength

TABLE 2 : CHEMICAL COMPOSITION OF THE MATERIALS STUDIED IN THE VALIDATION PROGRAM

Mark	C	Si	Cr	Mo	Mn	Ni	N
EL	0,030	1,15	21,85	2,72	1,06	10,30	0,039
CC	0,038	1,20	22,11	2,75	0,71	10,43	0,042
DI	0,050	1,17	20,70	2,58	0,80	10,20	0,045
EK	0,035	1,08	21,1	2,51	0,89	9,7	0,054

specimens (width W=50 mm, thickness B = 25 mm) and 0,4T-CT specimens with thickness reduced to 5 mm (W=20 mm, B=5 mm), called mini-CT. The specimens have initial side-grooves taking up 10% of total thickness.

The specimens are fatigue precracked using a maximum stress intensity factor smaller than 20 MPa√m for 1T-CT specimens and 15 MPa√m for mini-CT specimens. The original crack size at the end of precacking (a_0) is equal to 0,55-0,6 W. Then larger side grooves are machined (20% of total thickness).

Test method

Tearing resistance tests are performed with controlled load line displacement rate (0,4 mm/minute). In the case of 1T-CT specimens, either single or multiple-specimen techniques are being used. In the case of mini-CT specimens, only the multiple-specimen technique is applied. Load and load line displacement are measured with an accuracy better than 1%.

Analysis

J integral is computed according to relation (1) and crack length is derived either from compliance or from 9-point optical measurements.

$$J = \frac{\eta_0 U}{B_N(W-a_0)} \quad (1)$$

where $\eta_0 = 2 + 0,522\,(1-a_0/W)$
U : area under the load-displacement curve
B_N : net thickness

Validity criteria in terms of maximum J and maximum crack extension Δa are :
J_{max} = minimum value of $(W-a_0)\,\sigma_F/15$ and $B_N\,\sigma_F/15$
$\Delta a_{max} = 0,15\,(W-a_0)$
where σ_F is the flow stress.
A typical flow stress value at 320°C for aged materials is 450 MPa. In this case the criteria become :
J_{max}= 600 and 120 kJ/m^2 resp. for 1T-CT and mini-CT
Δa_{max} = 3 and 1,2 mm resp. for 1T-CT and mini-CT

The (J ; Δa) points are fitted with a power-law curve of the form $J = C\,\Delta a^n$. A mean value of parameter $J_{0,2}$ is defined at the intersection between the curve and a 0,2 mm offset line with a slope of $4\sigma_F$ (σ_F = flow stress). Mean values of parameters J_1 and J_3 are defined at the intersection between the curve and vertical lines at Δa = 1 and 3 mm (J_3 is computed only for 1T-CT specimens).

III VALIDATION PROGRAM

In order to check the relevance of tearing resistance tests using mini-CT specimens (0.4T-CT with reduced 5 mm thickness), a validation program was undertaken. Our major concern was the level of scatter of the J-Δa results, which we feared might be enhanced by reducing the affected volume of material : there are only one or two primary ferritic grains ahead of the crack front of mini-CT specimens, whereas they are five times as numerous for 1T-CT specimens [5].

The validation program consists of testing mini-CT specimens and 1T-CT specimens taken from the same materials in order to compare the results. All tests were carried out at the temperature of 320°C, which is the temperature of pressurized water reactors hot legs.

Studied materials

The materials are type CF8M cast duplex stainless steels, procured according to RCC-M specifications [6]. The cast products are heat treated at 1050-1150°C and waterquenched. Six different products are studied (Table 1). Their chemical compositions are given in Table 2 and their ferrite contents range from approximately 20 to 30%.

Experimental results

For each material, 12 mini-CT specimens (this number was chosen from considerations about experimental scatter [5, 7]) and at least 6 1T-CT specimens were tested. The J-Δa results are displayed in figure 1.

Several observations can be made about the J-Δa results :
- For all materials, the (J ; Δa) points obtained using mini-CT specimens are in good accordance with the points obtained using 1T-CT specimens.

- Scatter is large for both geometries. Although sometimes larger in the case of mini-CT specimens, it is generally of the same order.
- The cracks of a few mini-CT specimens strongly deviated from the side-grooves plane (the angle can reach 30°) : in this case the value of J is non-conservative and the corresponding data have not been taken into account (figure 1-D). SEM examinations of fracture surfaces and optical observations of sections after etching were performed on these specimens, as well as on specimens whose cracks had remained within the side-grooves plane. The observations on specimens with deviated cracks showed that the cleavage microcracks of ferrite have a non-zero angle with respect to the side-grooves plane (and also with respect to the load direction). On the contrary, the angle is null in the case of specimens whose cracks remain within the side-grooves planes. These observations are illustrated in figure 2, which shows secondary cleavage cracks located just under the fracture surface. These results seem to show that the direction of crack propagation can be influenced by the crystallographic orientation of ferrite. Also, experimental scatter could be partially attributed to the variability of ferrite crystallographic orientation in the specimens with respect to the direction of applied load : tearing resistance is all the weaker as principal ferrite cleavage planes become perpendicular to the force direction. The evidence of the important role of crystallographic orientations is in accordance with the work of P. Joly [7], who has shown the effect of ferrite crystallographic orientation on the ductility of smooth and notched axisymmetrical tensile specimens. He also highligthed the role of austenite orientation on the cleavage of ferrite, which we have not investigated in the scope of this study.
- In the case of plate DI aged 10 000 h at 400°C, two distinct populations of points are observed : one corresponding to mini-CT specimens having an equiaxed structure, the other a columnar structure. These two populations will later be analysed separately.
- The validity criterion J_{max} is exceeded by mini-CT specimens corresponding to the less embrittled materials. However, the J values remain representative of the valid ones obtained with 1T-CT specimens. The criterion on J seems too severe, so we will not take it into account for the analysis (following section).
- The validity criterion Δa_{max} is seldom exceeded. However, it can be noticed that the (J ; Δa) points corresponding to rather large crack extensions ($\geq$ 1,2 mm) tend to be situated in the lower part of the 1T-CT population of points. The criterion on Δa seems appropriate but without margin. This effect is probably due to the reduced thickness of the specimens, since plasticity is all the more extended as one approaches plane stress conditions. We will apply strictly the criterion on Δa in the analysis.

Analysis

The purpose of the analysis is to determine parameters $J_{0,2}$ and J_1.

Case of mini-CT specimens. As stated in part II, mean values of parameters $J_{0,2}$, J_1 and J_3 can be computed using a power law curve fit of all valid (J ; Δa) points. With a view to take account of the level of scatter, lower and upper bound values of the parameters can also be computed using lower and upper bound power laws, defined at minus or plus two standard deviations. An example of mean, lower and upper bound fits is given in figure 3.

This method, that will be called method A, was applied to the six tested materials. In some cases, the exponent "n" of the mean curve fit ($J = C\,\Delta a^n$) obtained on mini-CT specimens appeared to be rather low compared to the ones obtained on 1T-CT specimens. This effect can be attributed to the particular distribution of the (J ; Δa) points within the interval 0,2 - 1,2 mm. This observation lead us to define a criterion on exponent n : a value smaller than 0,20 will be rejected on the grounds that no such value was obtained with 1T-CT specimens (not only in this program but also in our general toughness database).

If exponent n is less than 0,20, an alternative method (method B) is proposed : $J_{0,2}$ and J_1 values are evaluated by fitting a power law on each (J ; Δa) point, the exponent being taken equal to an arbitrary value of 0,45 corresponding to an average value obtained with 1T-CT specimens. This method provides as many values of $J_{0,2}$ and J_1 as tested specimens : it is therefore again possible to compute mean, minimum and maximum values for each material (remark : in method B a vertical "blunting line" is taken for the computation of $J_{0,2}$ instead of a slope of $4\sigma_F$, the effect on the result being negligible).

Case of 1T-CT specimens. When the multiple-specimen technique is used, analysis is performed as for the mini-CT specimens, that is involving mean, lower and upper bound fits.

When the single-specimen technique is used, individual mean curve fits, as well as individual $J_{0,2}$, J_1, J_3 parameters, are computed for each specimen. Then average, minimum and maximum values are derived.

The results obtained for all materials after completion of analysis are displayed in table 3. The $J_{0,2}$ and J_1 parameters derived from mini-CT specimens are in good agreement with those derived from 1T-CT specimens.

IV APPLICATION TO AN IN-SERVICE ELBOW

Three samples of approximate dimensions 55x54x6 mm were taken from the outer skin of a hot leg 50° elbow in Gravelines 4 reactor. This elbow had remained in service for 86 898 h at the temperature of 323°C. The ferrite contents measured on the samples range from 33 to 35%. The chemical composition measured on one sample is given in table 4.

In each sample, four 0,4T-CT (with reduced thickness B≈5mm) specimens were machined and tested at 320°C in the same manner as described for the validation program. The J-Δa results are displayed on figure 4. Mean, minimum and maximum power law fits are superimposed. Four points out of twelve have been excluded to define these laws :
- the extreme right-hand one, because the criterion $\Delta a \leq 1,2$ mm is exceeded,
- three others because their cracks have strongly deviated from the side-grooves planes.

TABLE 3 : ANALYSED TOUGHNESS RESULTS OF THE VALIDATION PROGRAM

	0,4T-CT B=5mm		1T - CT	
	$J_{0,2}$ mean min max (kJ/m^2)	J_1 mean min max (kJ/m^2)	$J_{0,2}$ mean min max (kJ/m^2)	J_1 mean min max (kJ/m^2)
DI 700h- 400°C meth. A	183 149 225	239 197 289	143 117 180	260 222 288
CC 1000h- 400°C meth. B	42 21 63	87 43 131	38 25 67	77 54 103
EK 30000h- 350°C meth. A	79 49 129	107 67 1[illegible]1	78 58 107	110 82 148
EK 30000h- 325°C meth. A	140 107 185	220 171 282	144 99 177	235 166 278
DI equi 10000h- 400°C meth. B	79 52 107	164 108 220	60 35 72	108 128 134
DI colum 10000h- 400°C meth. A	41 38 43	82 77 87	49 48 50	95 93 97
EL 3000h- 400°C meth. B	39 17 60	80 36 124	41 19 65	76 49 99

TABLE 4 : CHEMICAL COMPOSITION OF GRAVELINES 4 ELBOW (WT%)

C	Si	Cr	Mo	Mn	Ni	N
0,033	1,18	21,0	2,58	0,73	9,2	0,033

TABLE 5 : TOUGHNESS RESULTS OBTAINED ON GRAVELINES 4 ELBOW

	Method of analysis	$J_{0,2}$ mean min max (kJ/m^2)	J_1 mean min max (kJ/m^2)
Elbow 86898 h - 323°C	A	82 62 109	128 98 166

Exponent n is equal to 0,308 : subsequently method A is applied for the computation of parameters $J_{0,2}$ and J_1. Flow stress is unknown because there is not enough material to machine tensile specimens. We have arbitrarily taken 500 MPa, which is an upper bound value at 320°C (thus conservative for the computation of $J_{0,2}$) according to our tensile characteristics database.

The computed $J_{0,2}$ and J_1 parameters are given in table 5. These results appear to confirm previous estimations performed through our predictive method, which consists of deriving impact strength from chemical composition and aging conditions, and of using correlations between impact strength and fracture toughness. The use of mini-CT specimens in the case of Gravelines 4 therefore contributes to validate the general methodology used for the justification of French primary circuit elbows. Other elbows are planned to be characterized in the same manner.

Remark. The question whether the elbow outer skin is fully representative of the whole component is currently being investigated. The first results tend to show that the properties of the material near the surface are similar to the ones at mid-thickness after sufficient thermal aging, which is the case of the studied materials.

V CONCLUSION

An experimental program was carried out to study the use of small 0.4T-CT specimens with reduced thickness to estimate the fracture toughness of embrittled cast materials. The results showed that such *mini-CT* specimens are relevant and guidelines for further practice are proposed. The main outcomes are the following :

- the J-Δa results obtained with mini-CT specimens are correctly centered around those obtained with regular 1T-CT specimens. Scatter is of the same order.
- the results corresponding to specimens whose crack extensions have strongly deviated must be rejected.
- cracking seems to be very dependant on the crystallographic orientation of ferrite in the tested zone of the specimen.
- it seems acceptable to consider as quasi-valid the J values exceeding the validity criterion J_{max}, but not the Δa values exceeding the maximum crack extension criterion.
- analysis is performed through mean, minimum and maximum power law fits at plus or minus two standard deviations, provided that the exponent n of the law is higher than 0,20. If not, a second method is proposed, which sets exponent n at a fixed arbitray value (frequently encountered) and defines a curve for each specimen individually. The application of this methodology leads to a good agreement between the $J_{0,2}$ and J_1 parameters obtained with mini and 1T-CT specimens.
- in view of the above items, a number of 12 mini-CT specimens tested according to the multiple-specimen technique seems appropriate for a correct determination of fracture toughness properties.

An in-service elbow at Gravelines 4 reactor has been characterized with 12 mini-specimens taken from its outer skin according to the above guidelines. The results confirm estimations performed previously through our predictive method and subsequently contribute to validate the general methodology used for the justification of French primary circuit elbows. Other elbows are planned to be characterized in the same manner.

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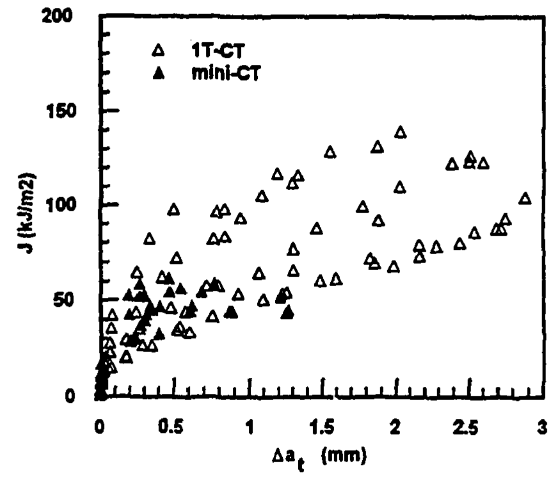


FIGURE 1-A: J-Δa RESULTS AT 320°C FOR MATERIAL EL AGED 3000H AT 400°C

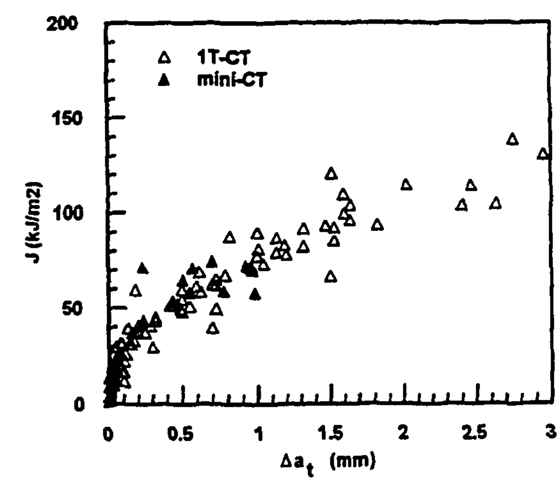


FIGURE 1-B: J-Δa RESULTS AT 320°C FOR MATERIAL CC AGED 1000H AT 400°C

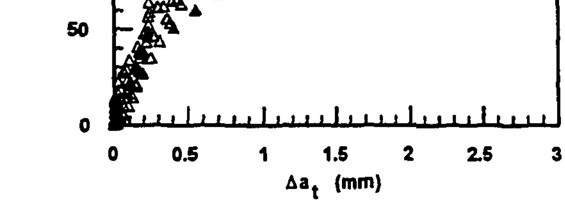


FIGURE 1-C: J-Δa RESULTS AT 320°C FOR MATERIAL DI AGED 10 000H AT 400°C

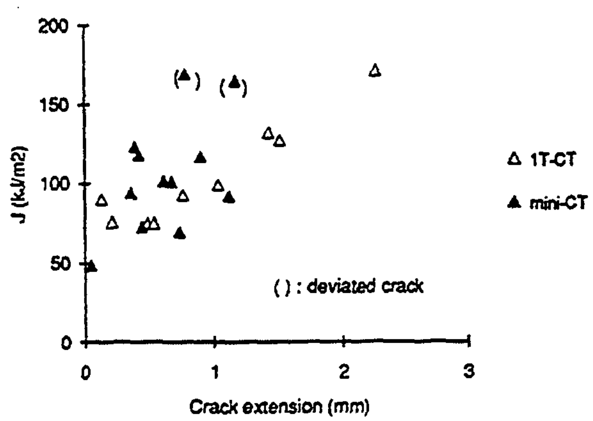


FIGURE 1-D: J-Δa RESULTS AT 320°C FOR MATERIAL EK AGED 30 000H AT 350°C

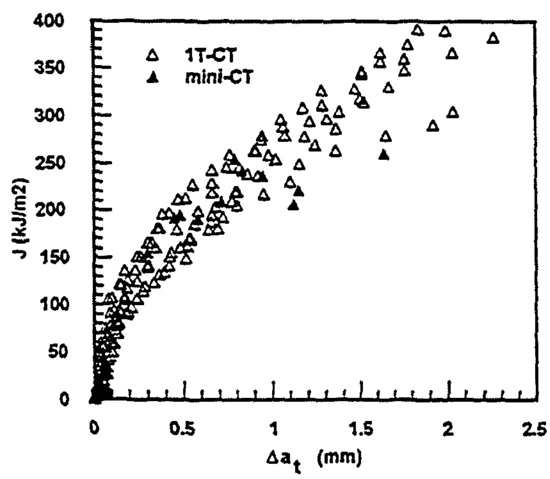


FIGURE 1-E: J-Δa RESULTS AT 320°C FOR MATERIAL DI AGED 700H AT 400°C

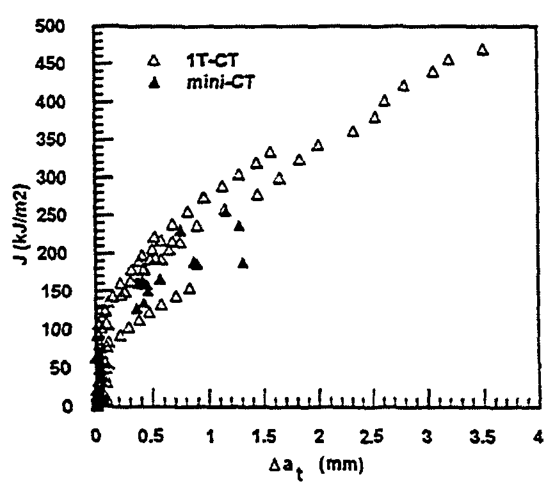
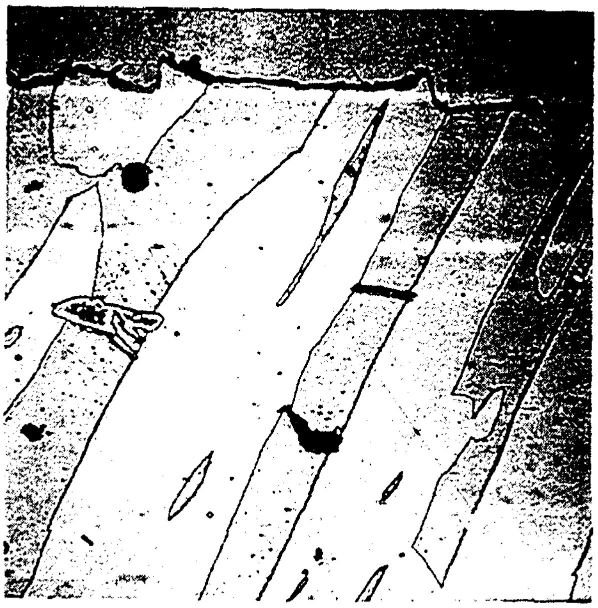


FIGURE 1-F: J-Δa RESULTS AT 320°C FOR MATERIAL EK AGED 30 000H AT 325°C



a) Specimen 1 : propagation along the side-grooves

b) Specimen 2 : propagation at 30° from the side-grooves

FIGURE 2 : FERRITE CLEAVAGE MICROCRACKS OBSERVED ON SECTIONS OF TWO MINI-SPECIMENS (X 400)

- VERTICALLY : direction of force
- HORIZONTALLY : direction of specimen notch
- OUT OF PLANE : direction of specimen thickness

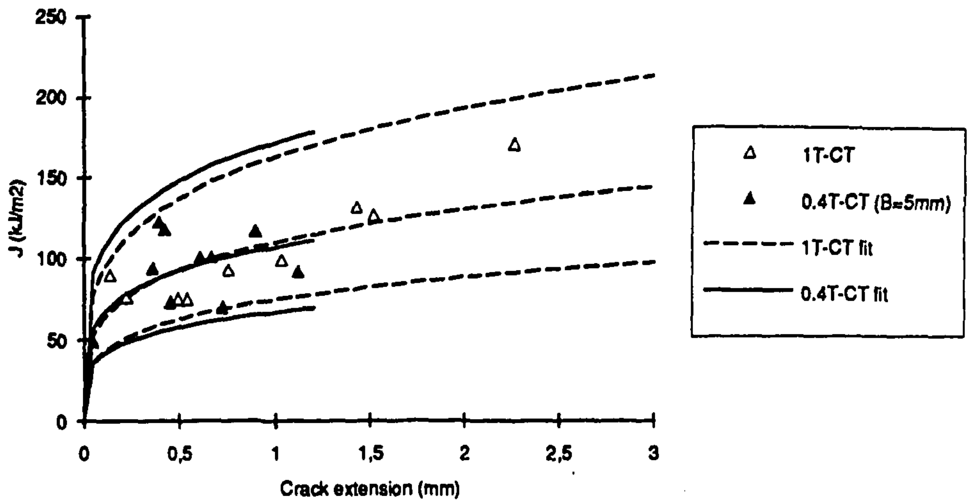


FIGURE 3 : POWER LAW FITS FOR MATERIAL EK AGED 30 000 H AT 350°C

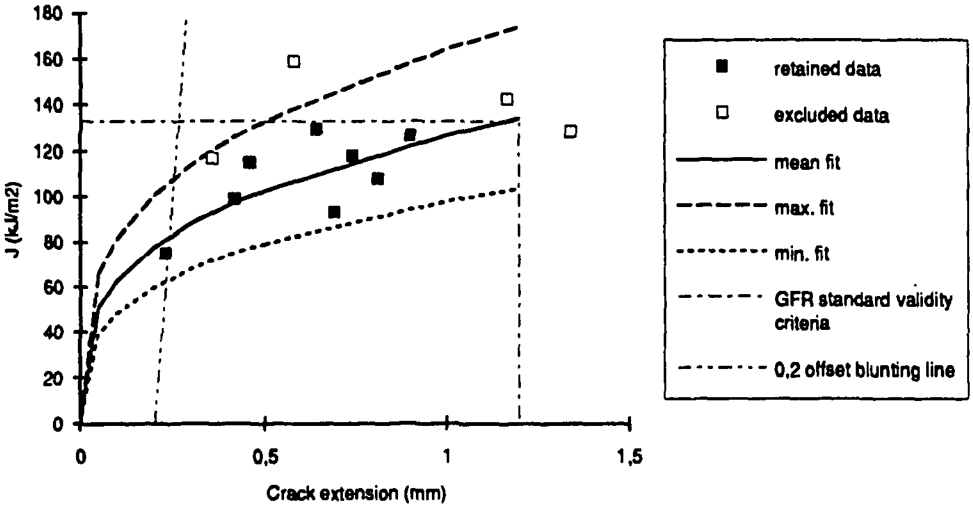


FIGURE 4 : J-Δa RESULTS AT 320°C FOR GRAVELINES 4 ELBOW USING MINI C(T) SPECIMENS