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Section 3.2. Small specimen technology

A remotely operated FIMEC apparatus for the mechanical characterization of neutron irradiated materials

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Abstract

From Flat-top Cylinder Indenter for Mechanical Characterization (FIMEC) test yield stress and tensile strength can be obtained. Results regarding different materials, most of them of fusion interest, show the general validity of the method. Moreover, indication on the ductile to brittle transition temperature (DBTT) of martensitic steels has been drawn by performing tests at different temperatures. FIMEC offers the possibility to perform several tests on a small volume of material, e.g. it has been estimated that 18 indentations can be made on a single disk ($\Phi = 25$ mm, $h = 5$ mm). For this reason, its application is of interest to characterize materials irradiated in the future IFMIF or in other sources with a limited irradiation volume. A remotely operated apparatus has been designed and costructed to work in hot cell on irradiated samples. It employs a WC punch ($\Phi = 1$ mm) and can operate at temperatures in the range between −180°C and +200°C. Details of the apparatus are presented.

1. Introduction

The Flat-top Cylinder Indenter for Mechanical Characterization (FIMEC) test is based on the penetration, at constant rate, of a flat punch of small size ($\Phi = 1$ mm, $h = 1.5$ mm). During the test the applied load (q) and the depth of penetration (δ) are measured, so load vs. penetration (LP) diagrams can be recorded. The characteristics of LP curves have been described in detail in previous works [1–4]: the limit pressure q_Y is reached after an initial linear stage, which is followed by a stage of work-hardening with loads tending to a saturation value q_S. In certain standardized conditions (penetration rate $\cong 0.0017$ mm s^{-1} and deformation rate in tensile test $\cong 10^{-3}$ s^{-1}), $q_Y \cong 3\sigma_Y$ and $q_S \cong 3\sigma_S$ (σ_Y and σ_S being yield stress and ultimate tensile strength, respectively).

FIMEC tests performed at various temperatures on ferritic and martensitic steels give an indication about the ductile to brittle transition temperature (DBTT) as obtained by Charpy impact tests employing standard V-notched specimens. FIMEC offers the possibility to perform several tests on a small volume of material [5,6] and can be of great interest for materials irradiated in neutron sources with extremely small irradiation zone. In this perspective, a remotely operated apparatus suitable for hot cell test has been designed and constructed. Details of its charateristics are reported in this paper.

Another object of this work is to verify the applicability limits and the sensitivity of FIMEC method. For this reason, tests have been performed on materials with different structural and mechanical characteristics: pure polycrystalline metals, light alloys, steels, metal matrix composites and particle reinforced alloys. Some materials have been examined after different thermal and mechanical treatments and at different temperatures. Some experiments were aimed to ascertain whether the method is sensitive to reveal directionality of mechanical properties. Most of the tested materials are of interest to fusion.

2. Experimental

The FIMEC apparatus set-up is shown in Fig. 1(a). The apparatus has been designed to be remotely operated by means of telemanipulators and remote connec-

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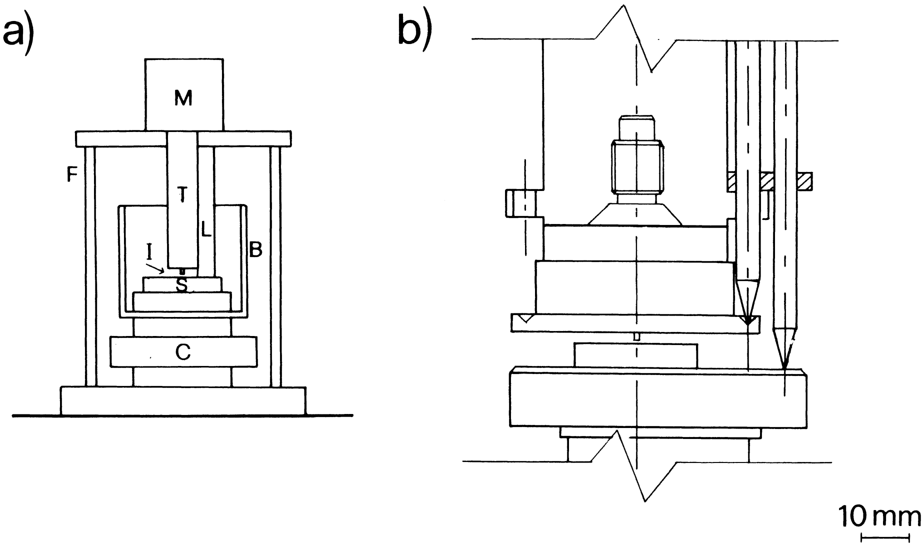

Fig. 1. (a) and (b); Set up of the FIMEC apparatus. (a): frame (*F*), stepping motor (*M*), translator rod (*T*), *L* (LVDT), indenter (*I*), specimen (*S*), load cell (*C*), box with thermostatic baths (*B*).

tions. A frame, dimensioned to have elastic deformation of the order of 1 μm in the maximum load condition (10 kN), supports the linear actuator, which drives the indenter advancement, the load cell, the LVDT displacement measuring system and the box for thermostatic baths.

The linear actuator is an electro-mechanical drive equipped with a stepping motor. The motor rotation is transmitted to a ball screw via a precision reduction gear; the ball screw converts the rotary motion at the gear output to translation, which is guided by means of a pre-loaded ballspline. The indenter is a flat punch of small size ($\Phi = 1$ mm, $h = 1.5$ mm) and is made of WC to guarantee good rigidity and non deformability. It is mounted at the end of the translator rod, whose advancement speed is constant and adjustable in the range from 0.0001 to 0.02 mm s^{-1}. The motor control unit is connected to an LVDT and a feedback system provides constant speed with applied load changes up to 10 kN.

The LVDT system measures the distance between specimen holder and indenter (Fig. 1(b)) with a resolution of 1 μm. The load cell, located under the specimen holder, has a resolution of 1 N. A picture of the FIMEC apparatus is shown in Fig. 2.

For tests at low and high temperature, the indenter and the specimen holder are contained in a box with thermostatic baths, allowing temperature changes in the range from −180°C to +200°C. Water or oil baths are utilized for tests at high temperature, ethanol-water solutions or liquid nitrogen for tests at low temperature. The operations of FIMEC apparatus, the on-line acquisition of load and penetration depth data and the graphic output are controlled by a specific software.

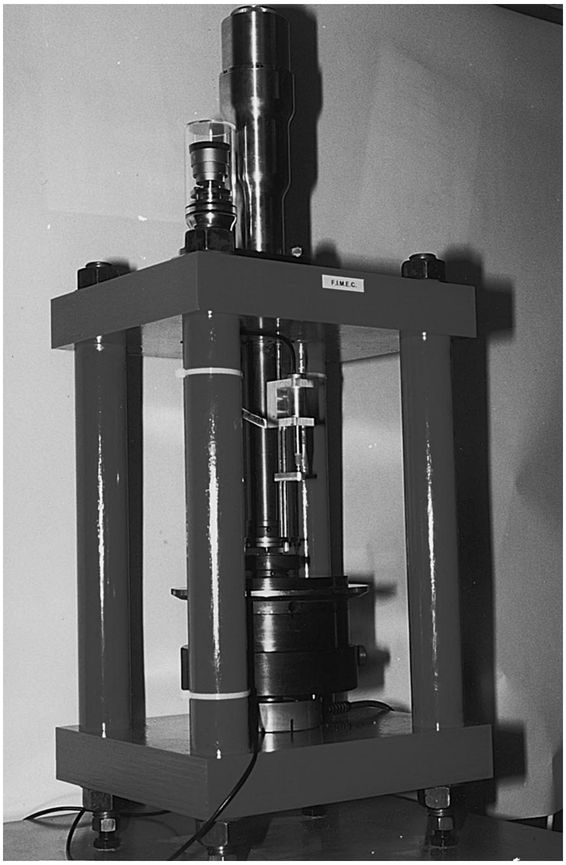

Fig. 2. FIMEC apparatus.

To control the position of each indentation and thus to optimize the space used for tests on the specimen, the specimen holder is mounted on a plate, which can rotate and translate. The indentation pattern shown in Fig. 3 permits 18 tests on a single disk with diameter of 25 mm and thickness of 5 mm. A distance of 5 mm between the centres of two adjacent imprints has been determined to be a minimum distance to avoid overlapping stress fields of neighbouring imprints, which can affect the LP curve trend [6]. For this reason, the circles in Fig. 3 have a diameter $\Phi = 5$ mm and not $\Phi = 1$ mm, which is the punch diameter.

FIMEC tests are normally repeated at least five times for each material, and the results show a good reproducibility (within ±3%). Data presented in this paper are the mean values. FIMEC data are compared with data from standard tensile tests carried out with a strain rate of 10^{-3} s^{-1}.

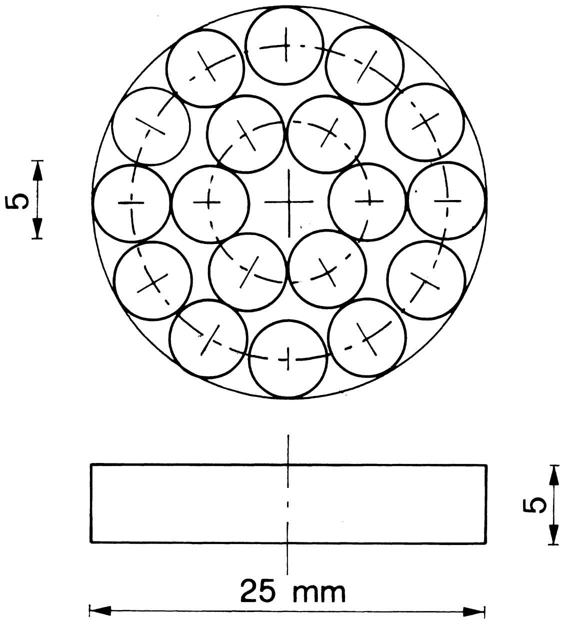

Fig. 3. Pattern of 18 indentations performed on a single disk ($\Phi = 25$ mm, $h = 5$ mm).

3. Results and discussion

The examined materials are listed in Table 1 along with the values of σ_Y yield stress from standard tensile

Table 1
Comparison between yield stress σ_Y obtained in standard tensile test and $q_Y/3$ obtained in FIMEC test.

Material	Tensile test	FIMEC test	Δ
	σ_Y (MPa)	$q_Y/3$(MPa)	
Al	60	55	0.08
Cu	190	185	0.03
Zn	55	55	0
Mo	540	560	−0.04
Al–7%Si–SiC	280	275	0.02
A986 deformed	35	32	0.09
Cu–5%Zn–7%Sn	145	127	0.12
GlidCop ([a])	483	420	0.13
Cu–0.65%Cr–0.08%Zr ([b])	306	285	0.07
Fe–40%Al + 1%Y_2O	922	980	−0.06
W + 1%La_2O_3 (⊥)	765	710	0.07
W + 1%La_2O_3 (//)	X	850	
TMZ	960	910	0.05
AISI 1040	450	425	0.06
MANET II	640	575	0.10
BATMAN-1951	510	440	0.14
BATMAN-1952	510	445	0.13
BATMAN-1954	490	470	0.04
BATMAN-1955	520	445	0.14
BATMAN-1953	520	510	0.02
BATMAN-1954 b	490	455	0.07
BATMAN-1956	510	500	0.02
F 82 H Mod.	520	490	0.06

$\Delta = (\sigma_Y - q_Y/3)/\sigma_Y$ (X not available).
[a] Glidcop (Cu–0.48Al_2O_3) deformed 20%.
[b] Elbrodur HF heated at 450°C.

test, of $q_Y/3$ from FIMEC test and the relative difference $\Delta = (\sigma_Y - q_Y/3)/\sigma_Y$. Δ values result to be always within the range ±0.15. Considering that the reproducibility of the σ_Y values, expressed by the C_N coefficient (C_N = standard deviation/mean value), is about 0.07 [7] and that the results of a tensile test are dependent on sample preparation, experimental apparatus or testing conditions, the $q_Y/3$ values are close to the σ_Y ones.

The specimen of W + 1% La_2O_3 used for FIMEC tests was a tile with strongly oriented grains (perpendicular to the base of the tile). Different $q_Y/3$ values have been obtained by performing tests on the base (//) and on the side face (⊥) of the tile showing that FIMEC test is able to detect anisotropy in σ_Y. Unfortunately, due to tile dimensions it was not possible to obtain standard tensile probes cut perpendicularly to the tile face and thus to compare in this case σ_Y and $q_Y/3$ values.

Table 2 compares results of FIMEC and tensile tests performed at +100°C on MANET steel after successive steps of thermal treatment at 700°C. The σ_Y value changes depending on the time of heating and the $q_Y/3$ value shows a similar trend. Δ values are substantially comparable for all the tests. Δ values tend to be a little higher in tests made at lower temperatures, but FIMEC test clearly shows the σ_Y evolution.

As previously mentioned, FIMEC tests can give an indication about the DBTT of ferritic and martensitic steels. In Fig. 4(a) and (b), the LP curves for MANET and BATMAN steel, obtained at different temperatures in the range between −180 and +100°C, are presented. For both the steels it is possible to distinguish two groups of curves corresponding to low and high test temperatures. The LP curves at low temperatures exhibit higher values of the work-hardening rate ($\Delta q/\Delta\delta$) in the non linear stage. Furthermore, slightly higher values of q_Y are observed. The transition between the two groups of curves occurs in a relatively narrow range of temperature. So the evolution of the $\Delta q/\Delta\delta$ work-hardening rate can be considered to give an indication on the ductile to brittle transition.

Fig. 5 shows average $\Delta q/\Delta\delta$ values plotted in reversed scale vs. test temperature and impact energy curves obtained in Charpy tests with ISO-V and KLST specimens. The transition temperature in FIMEC test ($DBTT_{FIMEC}$) has been determined as the temperature corresponding

Table 2
Results of tests performed at 100°C on MANET, cooled from $T = 1075$°C with a rate of $\dot{T} = 150$°C/min and then tempered at 700°C for the indicated times

Tempering time at 700°C (h)	Tensile test	FIMEC test	Δ
	σ_Y (MPa)	$q_y/3$ (MPa)	
2	600	613	−0.02
4	520	552	−0.06
6	600	600	0
12	560	573	−0.02

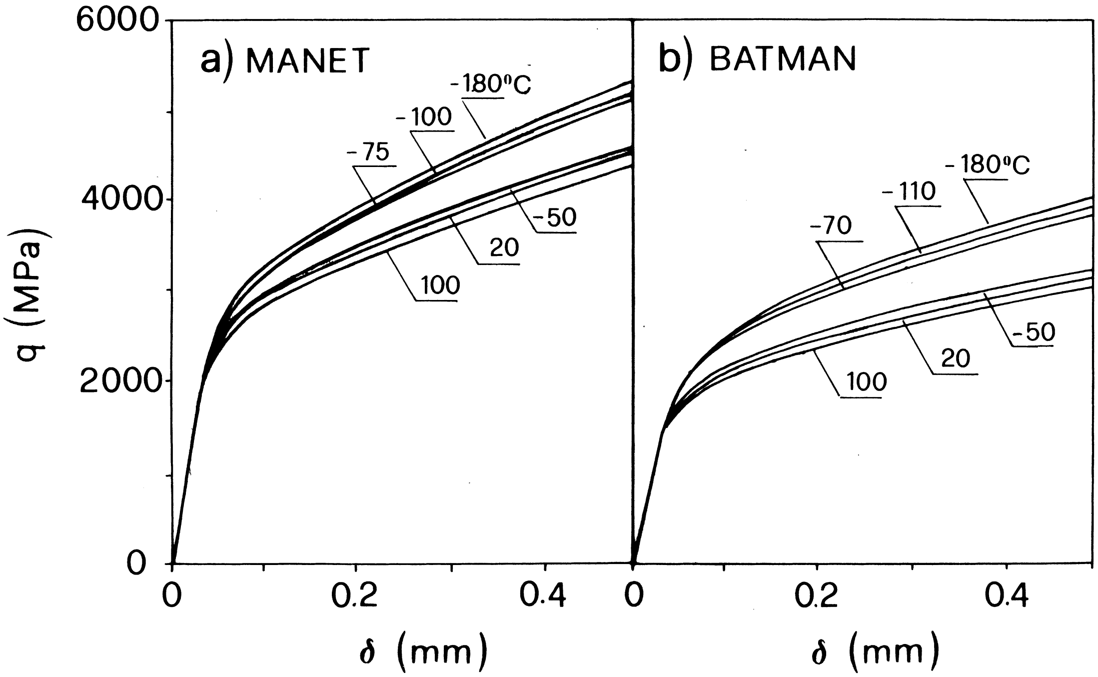

Fig. 4. LP curves of MANET (a) and BATMAN (b) steels obtained at different temperatures.

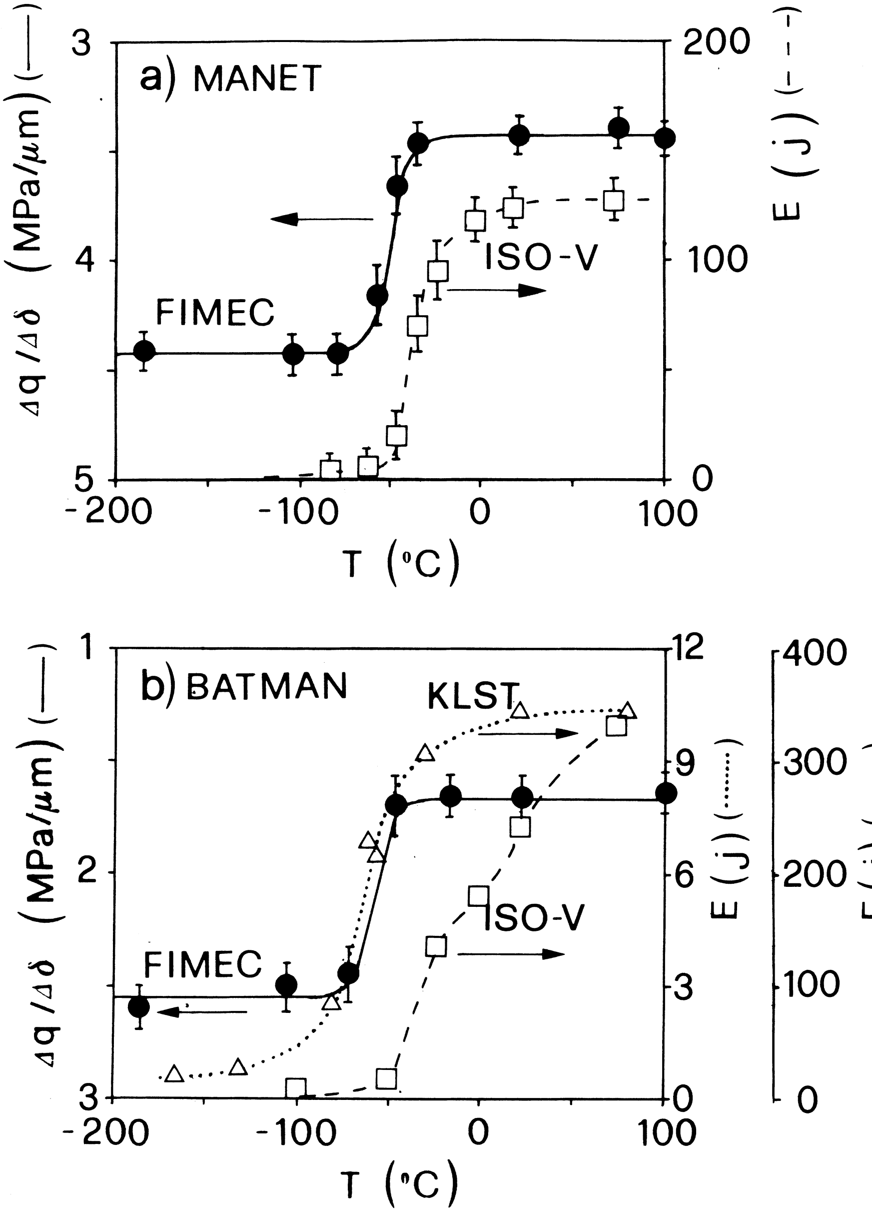

Fig. 5. Average values of $\Delta q/\Delta\delta$ work-hardening rate in the non linear stage are plotted in reversed scale vs. test temperature for MANET (a) and BATMAN (b) steel. ISO-V (a-b) and KLST (b) Charpy impact energy (*E*) are reported for comparison.

to the middle value between $\Delta q/\Delta\delta$ maximum and minimum.

Table 3 compares DBTT values from ISO-V and KLST Charpy tests and transition temperatures from FIMEC tests for steels of fusion interest. The ratios between the absolute temperatures, $T_{FIMEC}/T_{ISO\text{-}V}$ and T_{FIMEC}/T_{KLST}, are reported too. The ratio T_{FIMEC}/T_{KLST} is centred around a value of 0.95 with maximum variations of ±0.12; while the ratio $T_{FIMEC}/T_{ISO\text{-}V}$ is centered around 0.81 with a slightly higher data dispersion (±0.13).

4. Conclusions

The application of the FIMEC test appears of interest to characterize materials irradiated in neutron sources. In this perspective, a remotely operated apparatus was designed and constructed. The optimization of indentation pattern on the sample gives the possibility to perform 18 indentations on a single disk with a diameter of 25 mm and thickness of 5 mm, a robust geometry suitable for remote handling and manipulation. This number of tests is sufficient to determine yield stress,

Table 3
Comparison between DBTT values obtained in ISO-V and KLST Charpy tests and in FIMEC test

Materials	$DBTT_{(ISO\text{-}V)}$ (°C)	$DBTT_{(KLST)}$ (°C)	$DBTT_{(FIMEC)}$ (°C)	$T_{FIMEC}/T_{ISO\text{-}V}$	T_{FIMEC}/T_{KLST}
MANET II	−35	X	−50	0.94	X
Q + 4 h at 700°C	−5	X	−70	0.76	X
Q + 7 h at 700°C	0	X	−20	0.93	X
Q + 10 h 700°C	−10	X	−50	0.85	X
BATMAN-1951	0	−50	−85	0.69	0.84
BATMAN-1952	−15	−70	−85	0.73	0.93
BATMAN-1954	−5	−70	−55	0.81	1.07
BATMAN-1955	−10	−70	−95	0.68	0.88
BATMAN-1953	−30	−65	−80	0.79	0.93
BATMAN-1954 b	−15	−60	−70	0.79	0.95
BATMAN-1956	−15	−60	−55	0.84	1.02
F 82 H Mod.	−45	−80	−95	0.78	0.92

Quenched (Q).
(X not available).

ultimate tensile strength at different temperatures and to obtain indications about DBTT. Data regarding different materials, most of them of fusion interest, show the general validity of FIMEC test. Furthermore, this test is sensitive enough to monitor the evolution of mechanical characteristics following thermal treatments and to evidence a directionality due to a strong texture.

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