Available online at www.sciencedirect.com
ScienceDirect
Journal of Nuclear Materials 367–370 (2007) 648–652
journal of nuclear materials
www.elsevier.com/locate/jnucmat

High temperature indentation tests on fusion reactor candidate materials

R. Montanari [a,*], G. Filacchioni [b], B. Iacovone [a], P. Plini [a], B. Riccardi [c]

[a] *Dipartimento di Ingegneria Meccanica, Università di Roma-Tor Vergata, Via del Politecnico 1, I-00133 Rome, Italy*
[b] *ENEA CR Casaccia, Via Anguillarese 301, I-00060 S.M. di Galeria, Rome, Italy*
[c] *Associazione EURATOM-ENEA sulla Fusione, P.O. Box 65, I-00044 Frascati, Rome, Italy*

Abstract

Flat-top cylinder indenter for mechanical characterization (FIMEC) is an indentation technique employing cylindrical punches with diameters ranging from 0.5 to 2 mm. The test gives pressure–penetration curves from which the yield stress can be determined. The FIMEC apparatus was developed to test materials in the temperature range from −180 to +200 °C. Recently, the heating system of FIMEC apparatus has been modified to operate up to 500 °C. So, in addition to providing yield stress over a more extended temperature range, it is possible to perform stress–relaxation tests at temperatures of great interest for several nuclear fusion reactor (NFR) alloys. Data on MANET-II, F82H mod., Eurofer-97, EM-10, AISI 316 L, Ti6Al4V and CuCrZr are presented and compared with those obtained by mechanical tests with standard methods.

1. Introduction

FIMEC is an indentation technique employing a cylindrical punch [1–4]. Usually, a punch with diameter $\Phi = 1$ mm and axial length $l = 1.5$ mm is employed in the tests. However, depending on material characteristics and the size of the zone to be examined, punches of reduced (up to 0.5 mm) or larger size can also be used. During the test, applied load (L) and penetration depth (P) are measured; from L–P curves, it is possible to determine pressure (q) vs. penetration depth curves by dividing loads by the punch–surface contact area A.

After an initial elastic stage the FIMEC curves show three plastic stages. The first one is almost linear and ends at a pressure q_Y; the indentations show permanent sharp edges. For $q > q_Y$ the slope of the curve markedly decreases (second stage); the material starts to protrude around the imprint. Finally, the third stage shows an almost constant slope. Under certain conditions (penetration rate $\cong 1.66 \times 10^{-3}$ mm s^{-1}, deformation rate in tensile test $\cong 10^{-3}$ s^{-1}), $q_Y \cong 3\sigma_Y$, σ_Y being the yield stress ($YS_{0.2}$).

From FIMEC curves at different temperatures, knowledge of the DBTT (ductile to brittle transition temperature) can be gained [5,6].

* Corresponding author. Tel.: +39 06 72597182; fax: +39 06 2021351.
E-mail address: roberto.montanari@uniroma2.it (R. Montanari).

0022-3115/$ - see front matter © 2007 Elsevier B.V. All rights reserved.
doi:10.1016/j.jnucmat.2007.03.099

Table 1
Composition of tested materials (wt%)

Material	C	Cr	Mo	Ni	Mn	Zr	Nb	V	W	Ta	Ti	Si	Al	Cu	Co	N	S	B	Fe
MANET-II	0.10	10.37	0.58	0.65	0.76	–	0.16	0.21	–	–	–	0.18	0.007	0.01	0.005	0.032	0.005	0.007	Bal.
F82H mod.	0.09	7.67	0.01	0.02	0.16	–	0.0004	0.16	1.96	0.04	0.01	0.11	–	–	–	0.005	–	0.001	Bal.
EUROFER-97	0.10	8.87	0.03	0.028	0.45	–	0.0025	0.20	1.15	0.14	0.005	0.05	–	–	–	0.017	–	0.001	Bal.
EM-10	0.099	8.97	1.06	0.07	0.49	–	–	0.013	–	–	0.01	0.46	–	–	–	0.014	–	0.002	Bal.
AISI 316 L	0.02	17.34	2.40	12.50	1.80	–	–	–	–	–	–	0.32	–	–	–	0.080	–	0.001	Bal.
Ti6Al4V	<0.08	–	–	–	–	–	–	4.0	–	–	Bal.	–	6.2	–	–	<0.05	–	–	<0.25
CuCrZr-IG	–	0.6/0.9	–	<0.03	–	0.07/0.15	–	–	–	–	–	–	–	Bal.	–	–	–	–	<0.03

For its features, the FIMEC test has been widely used to investigate the evolution of local properties in the molten and heat affected zones of welded joints after post-welding heat treatments [7–9] and could be usefully employed to measure the mechanical properties of irradiated materials.

The FIMEC apparatus was originally developed and presented at ICFRM-8 [4] and was applicable for materials testing in the temperature range from −180 to +200 °C. Tests above room temperature were performed by heating the samples in an oil bath. Recently, the apparatus has been modified to operate up to +500 °C. So, in addition to providing yield stress at higher temperature, it is possible to perform stress–relaxation tests in a temperature range of great interest for several fusion reactor candidate alloys. To assess the validity of FIMEC results at high temperature, some fusion relevant materials (MANET-II, F82H mod., Eurofer-97, EM-10, AISI 316 L, Ti6Al4V and CuCrZr) have been tested and the data compared with those obtained by mechanical tests with standard probes. The compositions of the examined materials are reported in Table 1.

2. Experimental set-up

The experimental set-up is sketched in Fig. 1. A massive frame, rigid enough to show a total deformation of about 1 μm at the maximum applied load (10 kN), hosts all the components. The linear actuator is an electro-mechanical drive equipped with a stepping motor. The motor rotation is transmitted to a ball screw by a precision reduction gear; the ball

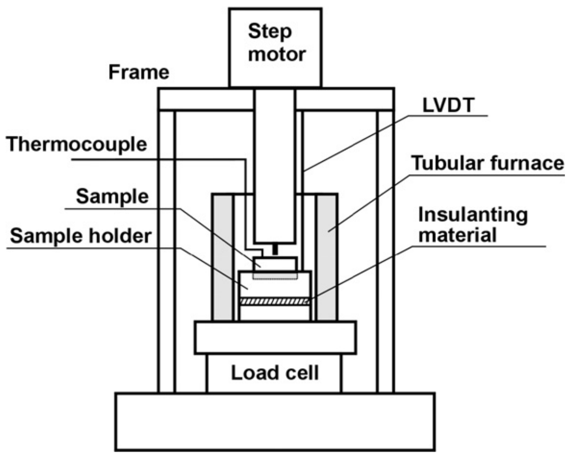

Fig. 1. Sketch of FIMEC apparatus.

screw converts the rotation at the gear output to translation, guided by means of a pre-loaded ballspline. The flat indenting punch is made of tungsten carbide, providing high rigidity and strength. The indenter is mounted at the end of a rod and the advancement speed can be varied between 1×10^{-4} and 2×10^{-2} mm s^{-1}. The motor control unit is connected to the displacement measuring system, a linear variable differential transformer (LVDT), and a feedback system provides constant speed in the full range of the load applied. The LVDT measures the displacement between the sample holder and the indenter with a resolution of 1 μm and the load cell, located under the sample holder measures the applied load with a resolution of 1 N.

The heating system consists of a tubular furnace, which guarantees a constant temperature (±2 °C) in a vertical zone of about 10 cm, where punch, sample holder and sample are located. Temperature fluctuations are ±1 °C in the central zone, where the sample is located. Disks of insulating material prevent overheating of the mechanical parts outside

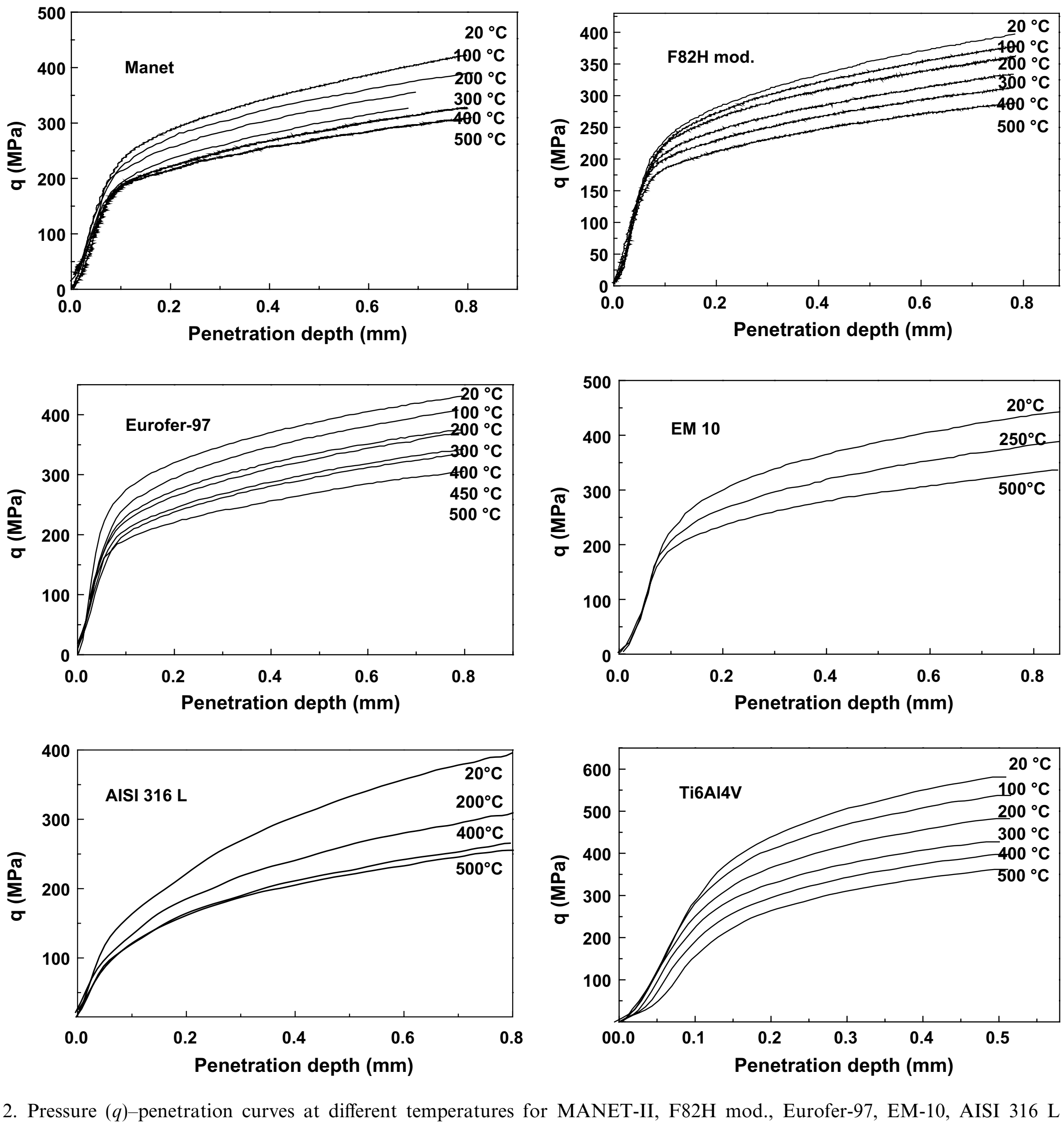

Fig. 2. Pressure (q)–penetration curves at different temperatures for MANET-II, F82H mod., Eurofer-97, EM-10, AISI 316 L and Ti6Al4V.

the furnace. The test temperature is measured by a thermocouple in direct contact with the sample. Indentation and sample heating are controlled by software specifically developed for the instrument.

To attain the thermal stabilisation of the samples, experiments are performed after a soaking time at the test temperature. Soaking duration depends on the test temperature and is of the order of a few minutes.

To perform stress–relaxation tests, penetration has been interrupted at a given point of the 2nd plastic stage of the *L*–*P* curve and load has been monitored for increasing time keeping constant the penetration depth, so load vs. time curves were obtained.

3. Results and discussion

Fig. 2 shows the FIMEC curves of tested materials. For each test temperature and tested material, five indentations have been performed and the results show a very good reproducibility. In practice, q_Y is obtained by observing the deviation (0.01 mm) from the straight-line portion (1st plastic stage) of the curve.

The average $q_Y/3$ values and standard deviations (S.D.) are reported in Table 2. For comparison, the corresponding average σ_Y values obtained by tensile tests with standard methods and the relative differences $\Delta = (\sigma_Y - q_Y/3)/\sigma_Y$ are included in the table. For all the examined materials the difference Δ never exceeds 7%, the same value of data scattering in tensile tests on the same material [10].

Fig. 3 shows an example of stress–relaxation tests performed on CuCrZr alloy at 20, 300, 400 and 500 °C. At each temperature, the indentation test has been interrupted in the 2nd plastic stage and load vs. time has been recorded while penetration depth was kept constant. Stress–relaxation consists of a progressive transformation of elastic strain ε_e to plastic strain ε_p at constant temperature. Since the total strain, i.e. the sum of ε_e and ε_p, remains constant, the stress (q) diminishes.

The results provide evidence that stress–relaxation strongly depends on temperature range. For example, the relative variation of q after 2×10^3 s is 0.05 at 20 °C, 0.06 at 300 °C, 0.10 at 400 °C and 0.21 at 500 °C. The tests reveal that stress–relaxation accelerates above 0.5 T_M where the physical mechanisms typical of high temperature plastic deformation become operative. Therefore, the method can be used to investigate them.

Table 2
Comparison between data of yield stress obtained from FIMEC tests and tensile tests with standard methods. $q_Y/3$ is the average value of 5 measurements, S.D. the standard deviation, Δ the relative difference

Material	T (°C)	σ_Y (MPa)	$q_Y/3$ (MPa)	S.D.	$\Delta = (\sigma_Y - q_Y/3)/\sigma_Y$
MANET-II [11]	20	655	640	10.81	0.02
	100	–	641	11.01	–
	200	607	639	10.52	−0.05
	300	602	595	9.35	0.01
	400	548	573	9.12	−0.05
	500	465	459	7.86	0.01
F82H mod. [12]	20	544	510	10.44	0.06
	100	501	488	8.89	0.03
	200	478	445	7.76	0.07
	300	469	435	6.66	0.07
	400	452	420	6.05	0.07
	500	407	378	5.63	0.07
EUROFER 97 [13]	20	546	585	7.02	−0.07
	100	507	488	6.97	0.04
	200	484	467	6.33	0.03
	250	477	488	6.21	−0.02
	300	470	490	6.54	−0.04
	350	461	445	5.76	0.03
	400	447	479	5.01	−0.07
	450	426	448	6.31	−0.05
	500	396	408	6.19	−0.03
EM 10 [14]	20	491	509	5.87	−0.04
	250	433	463	6.23	−0.07
	500	361	382	6.18	−0.06
AISI 316 L [15]	20	310	318	5.82	−0.03
	200	277	280	5.27	−0.01
	400	268	255	5.34	0.05
	500	235	220	4.03	0.07
Ti6Al4V [16]	20	895	904	11.03	−0.01
	100	800	807	11.23	−0.01
	200	720	747	11.75	−0.04
	300	645	671	9.41	−0.04
	400	550	569	9.02	−0.03
	500	510	548	8.64	−0.07
CuCrZr [17]	20	298	305	4.53	−0.02
	100	286	292	4.76	−0.02
	200	265	273	4.21	−0.03
	300	247	247	5.09	0
	400	–	226	5.27	–
	500	–	217	5.31	–

In Fig. 4 a detail of the q vs. time curve at 400 °C is displayed. Penetration was stopped at point A. After a sudden drop (A–B), the curve shows a slower decay indicating that a significant portion of the stress is lost in the early stages of the tests.

The measurements of stress–relaxation by penetration tests offer some advantages with respect to

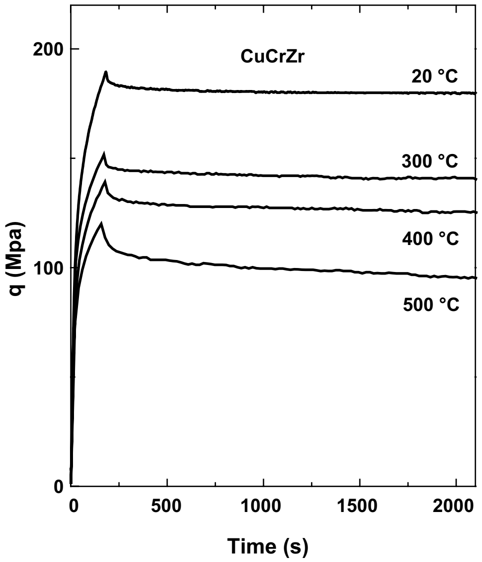

Fig. 3. CuCrZr alloy: pressure (q) vs. time curves recorded at 20, 300, 400 and 500 °C. When pressure drops the penetration has been stopped and the indentation depth is constant.

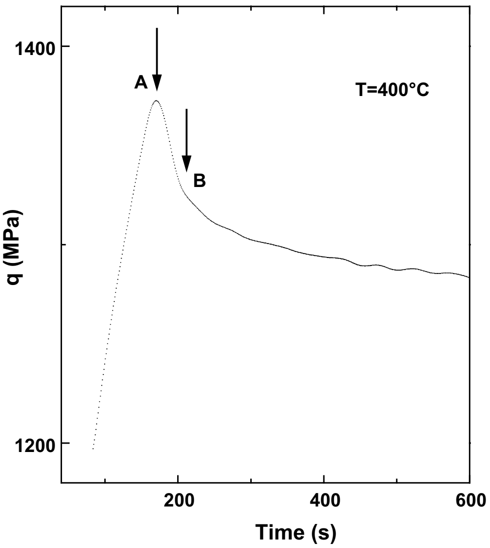

Fig. 4. CuCrZr alloy at 400 °C: after punch penetration has been interrupted (point A) pressure (q) exhibits a rapid drop to point B followed by a slower decrease.

traditional tests. In these latter tests, it is more difficult to keep the temperature constant along the probes and avoid spurious effects due to thermal gradients whereas in the case of FIMEC tests the indentation zone is relatively small and the test temperature is constant within ±1 °C. Furthermore, problems associated with slip of extensometer attachments are avoided.

4. Conclusions

The FIMEC apparatus developed by us permits to perform indentation tests up to 500 °C. MANET-II, F82H mod., Eurofer-97, EM-10, AISI 316 L, Ti6Al4V and CuCrZr have been tested at different temperatures and the results show that yield stress data from FIMEC are in good agreement with those from traditional tensile tests. Furthermore, the apparatus can be used for stress–relaxation tests at temperatures of interest for several fusion reactor candidate alloys. All test conditions can be achieved with ease; this represents an advantage with respect to analogous tests performed by means of tensile machines.

Acknowledgement

The authors are grateful to Dr Claudio Testani of C.S.M. (Centro Sviluppo Materiali – Castel Romano – Italy) who supplied Ti6Al4V alloy used in the present tests.

References

[1] R. Mouginot, D. Maugins, J. Mater. Sci. 20 (1985) 4354.
[2] H.Y. Yu, M.A. Imam, B.B. Rath, J. Mater. Sci. 20 (1985) 636.
[3] P. Gondi, R. Montanari, A. Sili, J. Nucl. Mater. 212–215 (1994) 1688.
[4] A. Donato, P. Gondi, R. Montanari, F. Moreschi, A. Sili, S. Storai, J. Nucl. Mater. 258–263 (1998) 446.
[5] B. Riccardi, R. Montanari, F. Moreschi, A. Sili, S. Storai, Fus. Eng. Des. 58&59 (2001) 755.
[6] B. Riccardi, R. Montanari, Mater. Sci. Eng. A 381 (2004) 281.
[7] S. Missori, R. Montanari, A. Sili, La Metall. Ital. 3 (2001) 35.
[8] G. Filacchioni, R. Montanari, M.E. Tata, L. Pilloni, J. Nucl. Mater. 307–311 (2002) 1563.
[9] R. Montanari, G. Filacchioni, B. Riccardi, M.E. Tata, G. Costanza, J. Nucl. Mater. 329–333 (2004) 1529.
[10] B.W. Christ, in: Metals Handbook, ninth ed., vol. 8. p. 32.
[11] L.O. Schafer, H. Kempe, MANET-II- Primärbericht, p. 11.
[12] F. Tavassoli, DEMO Interim Structural Design Criteria, Appendix A Material Design Limit Data, A3.S18F IEA F82H Steel, p. 18.
[13] F. Tavassoli, DEMO Interim Structural Design Criteria, Appendix A Material Design Limit Data, A3.S18E Eurofer Steel, p. 18.
[14] G. Filacchioni, ENEA CR Casaccia, private communication, 2002.
[15] G. Filacchioni, ENEA CR Casaccia, private communication, 2004.
[16] Boyer, Lampman (Eds.), Titanium Alloys Materials Properties Handbook, ASM, 2003.
[17] ITER Material Properties Handbook, MPH, ITER report S 74 MA2 98-04-14 W0.3, 1997.