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DEFECT DETECTION IN ENGINEERING CERAMICS USING DIFFERENT NON DESTRUCTIVE TESTING TECHNIQUES*

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Résumé - L'emploi des céramiques pour applications mécaniques à haute température et longue durée de vie nécessite des moyens de contrôle non destructif très performants. A cause des caractéristiques particulières de ces matériaux et de la faible taille des défauts recherchés, les techniques mises en oeuvre font l'objet d'un choix rigoureux. Leur application au contrôle industriel est discutée.

Abstract - The use of ceramics for high temperature and long lifetime applications require very sensitive non-destructive testing techniques. Due to the particular characteristics of these materials, and to the very small size of the flaws to be detected, they must be selected very strictly. Their application to industrial control is discussed.

I - INTRODUCTION

The interest in using ceramic materials to make parts of internal combustion or gas turbine engines grows more and more rapidly. Indeed, they contribute to reduce weight and fuel consumption, to increase combustion temperatures, and most of them are made from non-strategic raw materials.
Ceramics present major inconvenients which are a poor resistance to crack propagation and a lack of stress accomodation due to the absence of plastic deformation. For these reasons, crack-initiating defects are very small : a typical value for the critical defect size is 20 µm. They can be surface cracks, porosity, inclusions, voids, etc.. and may be generated during the forming process, firing, machining or transport.
Therefore, industrial fabrication of ceramic components with a high reliability need using appropriate non-destructive testing techniques, which must be easily implemented and offer good flexibility and adaptation to different shapes and sizes of parts.
For the present, this study concerns two structural ceramics : silicon carbide (SiC) and SiAlON, manufactured by CERAVER.
For this purpose, samples with representative flaws which can occur during the mass-production process have been made.

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II - TEST SPECIMENS (see for example ref./1/)

For SiC, three types of inclusions were choosen : alumina, iron and SiAlON. After firing (around 2100°C), only voids remain in some cases surrounded by slight structure perturbations. This phenomenon exists mainly for SiAlON and iron.

All the impurity sizes range from 30 to 250 µm. The sample thickness varies from 3 to 14 mm.
For SiAlON, only iron was used, in two sizes : 30-50 µm and 80-100 µm, with thicknesses of 3.8, 5.5 and 12.7 mm. The inclusions do not disappear after firing at 1750°C.

Material	Flaw nature	Specimen thickness (mm)	Specimen diameter (mm)	Flaw size (µm)			
				30-50	80-100	125-160	200-250
SiC	Voids	3	25	x	x	x	x
		4	38	x	x		
		5	25	x	x	x	x
		6	38	x	x		
		14	38	x	x		
SiAlON	Iron inclusions	3.8	36	x	x		
		5.5	36	x	x		
		12.7	36	x	x		

Table 1 - Test specimens

III - NON DESTRUCTIVE TESTING METHODS (see ref./2,3/)

The first purpose of this work is to determine which NDT methods could be applied to ceramic materials, and then to evaluate their limits in the optic of an industrial application.
It appears that three techniques can fulfill these conditions : X-Ray micro-radiography, fluorescent penetrant inspection and immersion ultrasonic testing. We also carried out experiments with an acoustic microscope, made by CGE (Marcoussis Laboratories) and valenciennes University.

1 - X-Ray microradiography (see ref./4/)

Microfocus X-Ray radiography was developed to obtain a better resolution on X-Ray images by reducing the geometric unsharpness, especially when using enlargement techniques. The focus spot size of these devices is smaller than 0,1 mm (usually 10 to 25 µm).
Due to the small flaws to be detected, using low kilovoltage (down to 25 kV) is a way to improve both contrast and resolution, because the generated X-Rays have longer wavelengths. But in this case, the most important problem is the low intensity of the radiation.
An other way to increase contrast is to get another radiation energy spectrum by choosing an adequate target material which emits longer wavelength X-Rays ; this solution does not require using lower kilovoltage, so the exposure time is much smaller (testing can be performed at 50 kV).

The first tests were made on seven microfocus radiography systems with low-sensitive, fine-grained films, without enlargement.
Some other experiments were made on a conventional X-Ray device, with a 3x3 mm focus spot size, which can operate at very low kilovoltage because of the high radiation intensity. It was possible to obtain very good images of the specimens, from 20 kV (3 mm thickness) to 40 kV (14 mm thickness). This is an adequate method to work on long focus-to-film distance, without enlargement, especially for the thickest parts which require a higher intensity radiation.

2 - Liquid fluorescent penetrant inspection

This method is very effective to detect very small surface flaws such as cracks, and is employed for all the critical parts of gas turbines for example.
It is difficult to realize artificial defects to determine the limit of detection of this technique on ceramics : one way is to make fine marks with a Vickers or Knoop penetrameter, but the low K1C of these materials cause a very complex cracks network which is not representative of a real flaw. Nevertheless, such indentation cracks were made on polished samples, under different loads, to obtain cracks from 50 to 900 µm long.
Three penetrants were tested, with different sensitivities.
The surface condition is a very important parameter ; it is very difficult to remove the excess penetrant on the surface after immersion : for green-machined or as-fired parts, the remaining penetrant can mask some flaws during examination. To solve this problem, water-washable penetrants can be used, but they are less sensitive than the post-emulsified ones, which can be employed on machined surfaces.

3 - Immersion ultrasonic testing

Ultrasonic examination is commonly used in many non-destructive testing applications. Immersion testing (in water) is the most reliable technique and offers many advantages : lack of contact with the part, constant transducer-to-piece distance, complex-shape components testing, higher sensitivity, precision of scan plans, etc. The usual frequencies are 5 or 10 MHz, with focused or non-focused transducers.
Adaptation of this technique to ceramics is difficult because of the ultrasonic characteristics of these materials (see table 2), and of the very small flaw sizes.

Material	Longitudinal waves velocity VL (m/s)	Shear waves velocity VS (m/s)	$\frac{VS}{VL}$	Acoustic impedance ($kg.m^{-2}.s^{-1}$)
SiC	11920	7580	0.64	$3.75.\ 10^{-7}$
SiAlON	10530	6000	0.57	$3.37.10^{7}$
Inconel 718	5810	3100	0.53	$4.76.10^{7}$

Table 2 - Compared acoustic properties of ceramics and Inconel 718 Superalloy.

The high longitudinal wave velocity creates front echo enlargement, so that it is impossible to control thin parts (less than 5 mm thick) with a good sensitivity. The use of shear waves does not seem to be a good solution because they generate more structural noise and thus decrease the minimum detectable flaw size.
Furthermore, the high velocity may also decrease the detection capability due to the increased wavelength. As an example, the wavelength in SiC at 10 MHz in the

longitudinal mode is 1.19 mm : this has to be compared to the flaw sizes (< or = 50 µm), supposing that the smaller the wavelength, the higher the detection capability.
One way to reduce this is to work at higher frequencies, 20 or 25 MHz, which may be the maximum value to have a good penetration in the case of thick parts (15 or 20 mm).
It's also necessary to improve signal-to-noise ratio, by using focused transducers with a short water-gap and a small focal spot size, which is commonly measured on a small ball at an amplitude level of - 6 dB.
The transducers used for this work are wide-band pulser-receivers, whose characteristics are given in table 3.

Frequency (MHz)	Active area diameter (mm)	Preferred water-gap (mm)	Focal beam diameter (-6dB) (mm)	Useful range in water (mm)
10	9.8	70	1.4	57
20	6.3	53	1.1	37
25	3.2	25	< 0.5	to be measured

Table 3 - Characteristics of ultrasonic transducers

The ultrasonic pluser-receiver has a wide-band frequency domain (up to 35 MHz), and is connected to a spectrum analyser (0 to 40 MHz) and a digital 100 MHz oscilloscope, to obtain both frequency and time-domain contents of the acoustic signals (see fig. 4).

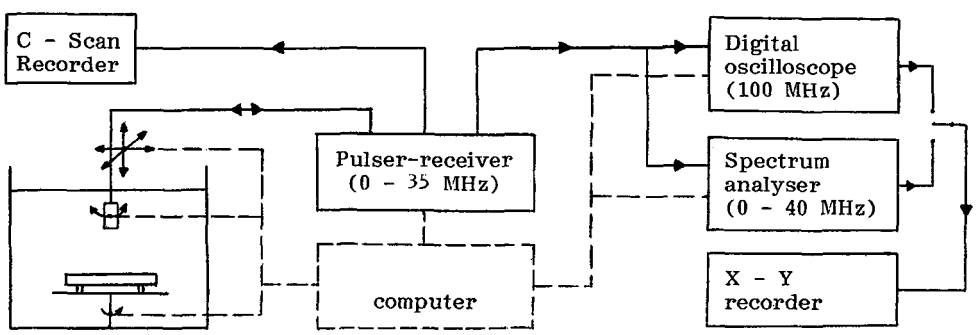

Fig. 4 - Sketch of the ultrasonic facility.

The whole mechanical system and the different appliances can be computer-assisted by a PDP 11/73.
The ultrasonic testing of the test samples shows that the detection capability is improved when the test frequency increases, but thin specimens inspection is not successfull.

4 - Acoustic microscopy (see ref ./5/)

This technique is based on the principle of ultrasonic pulser-receiver immersion testing, but operates at a 100 MHz frequency and requires special fine-focused transducers with a very short water-gap. The maximum depth that can be inspected with optimal sensitivity is 8 mm, and the limit of detection is about 10 µm.
The examination is performed following two operating modes : defect detection, then

defect imaging, with a computerised treatment of the acoustic response of the sample, which gives acoustic C-scan images on a TV Monitor.
Presently,this device operates only on flat parts of small areas (10 x 10 cm).
The experiments made on the thinest specimens showed that all defects, even porosity, were detected.

IV - RESULTS AND DISCUSSION

Table 5 summarizes the results obtained with the different non-destructive examination techniques.

NDT Techniques	Detected defects (in SiC or SiAlON) (µm)						
	Voids				Iron inclusions		Surface cracks
	30 - 50	80-100	125-160	200-250	30-50	80-100	
X-Ray microradiography	ND	D	D	D	D/ND	D	-
	Thickness < or = 6 mm						
X-Ray radiography	ND	D	-	-	ND	D	-
	Thickness > or = 6 mm						
Liquid fluorescent penetrant inspection	-	-	-	-	-	-	> or = 150 µm long on machined surfaces
Immersion ultrasonic testing	D/ND	D	D	D	D	D	-
	Thickness > or = 5 mm						
Acoustic microscopy	D	D	D	D	D	D	-
	Thickness < or = 9 mm						

Table 5 - Limit of detection of non-destructive testing techniques for ceramic materials.

It is shown that all these techniques have to be used as complementary NDT methods :
- For surface flaws detection (cracks or small voids), liquid fluorescent penetrant inspection is very sensitive, but surface condition is very important ;
- For internal flaws, such as voids or inclusions, X-Ray microradiography is a good technique in the case of thin parts, an can be coupled to a low-kilovoltage conventional X-Ray tube for thicker specimens (> or = 6 mm). Immersion ultrasonic testing must be performed at frequencies equal to or higher than 20 MHz, but it is not efficient for thin samples, thus being complementary to X-Ray inspection. Acoustic microscopy is the best technique to detect the smallest flaws, but its use is presently limited to laboratory examinations.

V - CONCLUSION

This study is in progress to improve each of these NDT techniques :
- X-Ray microradiography : using the enlarging technique on films, and research on other target materials.
- Liquid fluorescent penetrant inspection : improving standards to measure the limit of detection for all surface conditions.
- Immersion ultrasonic testing : this technique requires the highest developments on many points :
  . near-surface defect detection, with adequate transducers (near-surface and internal flaws detection will be considered separately),
  . accurate flaw size measurement and flaw nature determination from time and frequency domains contents of the ultrasonic echoes.

All that techniques already appear to be well-suited to an industrial non-destructive testing of ceramic materials. Further work will basically consist in reaching detection capability consistent with the specified maximum defect size , particularly in the case of surface or near-surface flaws.
On the other, the acoustic microscope is a very efficient laboratory device, but it must now be designed for industrial NDT.

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