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SPALLATION AND SHOCK-WAVE BEHAVIOUR OF SOME CERAMICS⁽¹⁾

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Résumé - L'utilisation optimum des céramiques en technologie des hautes pressions dynamiques nécessite non seulement de connaître certaines caractéristiques dynamiques comme la limite élastique d'Hugoniot et la résistance à l'écaillage, mais également les micromécanismes associés à leur déformation et à leur rupture. Ce papier fait le point sur ces différents aspects pour trois classes de céramiques : les verres de silice, les vitrocéramiques et les alumines frittées. Pour les verres, l'influence de la densification permanente de la silice sur la rigidité du matériau et sur sa résistance à l'écaillage est discutée. En ce qui concerne les vitrocéramiques, on montre l'existence d'une microfissuration du milieu pour une contrainte inférieure à leur limite élastique d'Hugoniot. Pour les alumines, on discute du rôle respectif de la microplasticité des grains et de la microfissuration de la phase vitreuse intergranulaire sur le dédoublement de l'onde de compression.

Abstract - To determine the best use of ceramics in shock-wave technology it is necessary not only to know certain dynamic characteristics like the Hugoniot elastic limit and the spall strength, but also the micromechanisms associated with their deformation and failure. This paper is a discussion of these different aspects for three types of ceramics : silica glasses, glass-ceramics and sintered alumina. For glasses, the influence of silica's permanent densification on the material's rigidity and on its spall strength is discussed. For glass-ceramics, we show the existence of a microcracking of the material for a stress lower than their Hugoniot elastic limit. For alumina, we discuss the respective rôle of grain microplasticity and of microcracking of the intergranular glassy phase on the formation of two-wave structure.

1 - INTRODUCTION

The dynamic behaviour of high-strength ceramics is of interest because of the expanding use of these materials in high pressure and shock-wave technology. Their optimum use demands not only knowledge of certain dynamic characteristics, like the Hugoniot elastic limit and spall strength, but also the micromechanisms associated with their deformation and failure. Our intention here is to discuss these different aspects for three types of ceramics : silica glasses, glass-ceramics and sintered alumina. There have already been different studies on these materials. In this paper we recall the principal results for each type of material and we discuss the different interpretations possible. We offer an original contribution in the discussion of the micromechanisms at the origin of irreversible deformation of glass-ceramics and alumina.

For each type of material, this study is limited to a Hugoniot stress equal to twice the Hugoniot elastic limit. So for silica glasses, glass-ceramics and alumina, the maximum stress in experiments is respectively about 17 GPa, 14 GPa and 15 GPa.

2 - BEHAVIOUR OF SILICA GLASSES

2.1. - Spallation

Spall damage is a type of fracture produced when a large tensile stress develops in

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material bodies through the interaction of stress-waves. Experimental investigations of spallation usually involve impacting a thin slab of the material of interest (the impactor) against a thicker slab of the same material (the target). Experimental data of spallation of silica glasses appear in two studies : one by G.I. Kanel et al. /1/ on a K-8 glass, and the other by J. Cagnoux /2,3/ on a Pyrex glass. These authors state :

- Below the Hugoniot elastic limit (HEL) spall strength increases with the velocity of the impactor (or with the impact stress), and the thickness of the sample.
- Above the HEL, there is a high spall strength (3 to 6 GPa) which decreases with the impact stress.

These results are reproduced on Figure 1. The interpretation of the responses observed can be the following.

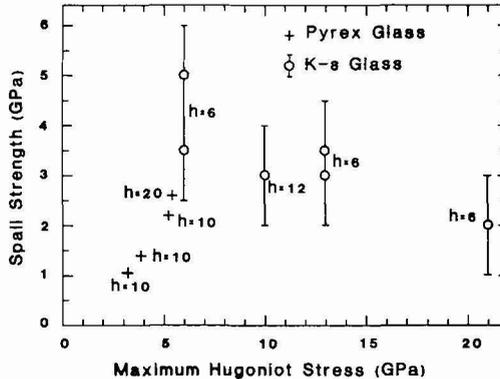


Fig. 1 - Spall strength versus maximum Hugoniot stress (or impact stress) for two types of glass. Pyrex Glass data are from Cagnoux (1982, 1985) et K-8 Glass data are from Kanel' et al. (1977). h is the thickness of the sample (in mm).

2.1.1. - Spallation of Pyrex glass below the HEL

We assume the existence of a stage of nucleation of microcracks, at the boundaries between the glassy microphases and the glassy matrix /2,3/, since Pyrex is a phase-separated glass.

The increase in velocity of the impactor induces the increase in the tensile stress in the interior of the target. As this tension is produced with a high strain-rate, the damage due to the growth of the biggest microcracks which are activated first, does not have time to relieve the stresses before smaller microcracks are activated in their turn. This delay in the stress-relieving leads to increase in the apparent spall strength. On the other hand, in Pyrex glass the release-waves form a shock-wave /3/ ; the increase in the strain-rate is then in proportion to the thickness of the sample. According to Kipp and Grady's model /4/ an increase in strain-rate entails a delay in the activation of microcracks, delaying the stress-relieving. In both cases (increase in the tensile stress and increase in the strain-rate) the delay in the stress-relieving gives rise to the activation of a greater number of flaws, which reduces the free path of microcracks, and the time to fracture.

2.1.2. - Spallation of K-8 glass above the HEL

Above the HEL, yielding of silica glasses is due to permanent densification (cf. 2.3.1. and 2.3.2.) and we will see in paragraph 2.3.3. that this phenomenon is accompanied by a production of heat. Just as this heating leads to a loss of rigidity of the glass, it also leads to a decrease in spall strength. We should note the similarity between the data on Figures 1 and 2.

2.2. - Shock-wave propagation

2.2.1. - General behaviour of fused quartz

In general, elastic moduli of substances increase with density. However, the bulk modulus of fused quartz decreases with pressure up to 2.3 GPa /5/. It has been reported /6,7/ that the wave propagation velocity decreases with increasing longitudinal stress up to about 3 GPa, and that a ramp precursor wave was observed. Fused quartz behaves anomalously not only in the elastic region, as above, but also in the more highly compressed region. Bridgman and Simon /8/ have reported a permanent increase in density of fused quartz statically pressurized to over 10 GPa. The effect of temperature and shear stress as well as pressure on this permanent increase in density has been studied by other investigators /9,11/. These studies indicate that the permanent densification occurs as the result of a rearrangement of the SiO₄ tetrahedra, without any crystallization and / or changes in the oxygen coordinations of the silicon atoms. The response to a shock-wave shows that in such an experiment the permanent densification starts at 8.81 GPa, value of the Hugoniot elastic limit. The tetrahedral coordinations seems to remain at least up to 22 GPa, but above 16 GPa, they would be distorted or in some parts destroyed. The pressure of 16 GPa is considered as the limit of the permanent densification process /12/.

2.2.2. - Behaviour of other silica glasses

The presence of network-modifying cations does not change the general behaviour described above for fused quartz, but lowers the permanent densification threshold /13/. On the other hand, among glasses presenting a phase separation, Pyrex glass was chosen for special study /3/. A Hugoniot elastic limit of 7.5 ± 0.5 GPa was found for this glass. Below this limit, the elastic properties are identical to those of fused quartz. Above this limit, permanent densification was clearly observable. Maximum densification was reached for a stress of around 17 GPa, a value comparable to 16 GPa given for fused quartz. After a loading of more than 17 GPa, the permanent densification obtained following unloading was lower than that obtained after a weaker amplitude loading. Similar results were obtained /13/ on glasses with different chemical compositions. This phenomenon has been interpreted as an annealing of the material provoked by the high temperature reached on the Hugoniot.

2.2.3. - Rigidity of glasses above the HEL

J. Cagnoux /3,15/ has used piezoresistive gauges to measure the deviatoric stress and the mean stress at the passage of a shock-wave. These results are reproduced on Figure 2. On this, we can see the approximately constant value of the deviatoric stress beyond the HEL, that is in the permanent densification region. This result had been interpreted /3/ as a softening of the glass due to the high temperature accompanying the permanent densification. Kondo et al. /16/ evaluated the following temperatures on the Hugoniot of fused silica : 710°K at 9.5 GPa, 1180°K at 15 GPa, 1920°K at 22 GPa. In addition, according to Spinner /17/, for a borosilicate glass, the shear modulus varies very little up to about 600°K, then decreases to lose about 35 % of its initial value at 900°K. This is approximately the temperature obtained in experiment D3 (Figure 2). Taking on initial shear modulus of 20 GPa at the HEL, the deviatoric stress calculated for the shot D3 is 3.8 GPa, which corresponds to the experimental value (Figure 2).

Certain authors /1,18/ believe that silica glasses fracture above their HEL. It seems that this interpretation should not be upheld for the following arguments :

- spall strength is non zero above their HEL (Figure 1),
- loss of rigidity in glasses above their HEL (Figure 2) should be correctly interpreted as a thermal softening of the material,
- the optic transparency of fused silica is maintained at least for a Hugoniot stress to 30 GPa /19/.

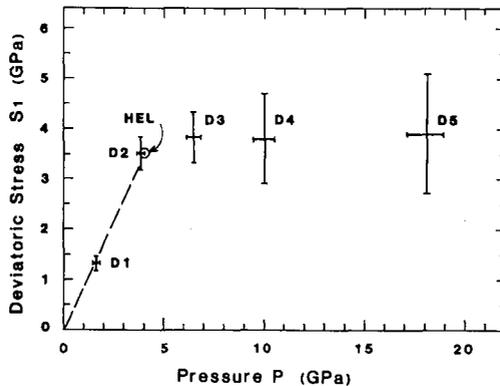


Fig. 2 - Deviatoric stress in the longitudinal direction versus pressure, from Cagnoux (1981, 1985).

3 - BEHAVIOUR OF GLASS-CERAMICS

Certain glass-ceramics have been studied in the past, particularly for their property of forming a ramp precursor /20,21/ ; but there is not to our knowledge a complete study on the shock-wave behaviour of these materials. We begin a such a study here with two glass-ceramics made by the Société Corning. These two glass-ceramics are named Vitro A and Vitro B and are 90 % crystalline phase (β quartz for Vitro A and β spodumene for Vitro B). Crystalline size is 0.05 μm for Vitro A and 2.5 μm for Vitro B.

3.1. - Experiments

Two types of experiments were performed, using a 110 mm-diameter compressed gas gun. The first were plate-impacts with wave profile measurements made by a VISAR interferometer or using piezoresistive gauges. For the two glass-ceramics, we measured :

- i) the Hugoniot Elastic Limit (HEL)
- ii) the spall threshold : the velocity of the flyer was progressively increased to observe the pull-back signal of incipient damage under tension,
- iii) the spall strength after loading up to the HEL.

The second type of experiments were plate-impact recovery. Both flyer and target were star-shaped as recommended by P. Kumar and R.J. Clifton /22/ to ensure a total trapping of radial release-waves. Three shots were performed for each material :

- i) one spallation type experiment with loading just above the spall threshold,
- ii) two loading-release experiments with maximum stress of about 0.2 and 0.6 times the HEL.

The description of all the experiments is given in Ref. 23.

3.2. - Results of spallation experiments

The results obtained on 100 mm thick targets are the following :

- The spall threshold of glass-ceramics A and B is respectively 0.7 and 0.5 GPa,
- The spall strength decreases when the impact stress reaches 0.5 x LEH,
- We recovered powder for glass-ceramic A and fragments for glass-ceramic B.

Examination of the latter indicates that spallation occurred in a single plane.

3.3. - Shock-wave compression : results and discussion

The stress-histories recorded for each of the glass-ceramics are shown on Figure 3. There is a similarity between glass-ceramic A's precursor and that of Pyrex glass. On the other hand, glass-ceramic B produces a stress ramp over the whole precursor. We cannot explain the difference between the precursors in these two glass-ceramics. The maximum stress reached by this precursor (about 8 GPa for the two glass-ceramics A and B) leads us to suppose that the two-wave structure is due to the permanent densification of the glassy phase. However the difference in speed between this precursor and the plastic-wave is much bigger for glass-ceramics than it is for silica glasses (Figure 3). Only damage in the material can be responsible for such a reduction in the plastic-wave speed in glass-ceramics. This is a hypothesis that we shall try to validate with the aid of observations made on recovered samples and of spallation experiments results.

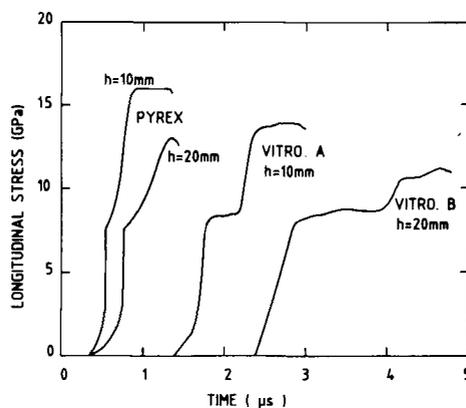


Fig. 3 - Stress-histories recorded in Pyrex Glass (Cagnoux, 1985) and in glass-ceramics A and B (Longy, 1987). h is the distance of propagation of the stress-wave.

After a loading-unloading cycle, we recovered :

- powder for glass-ceramic A after an impact stress of $0.2 \times \text{HEL}$, and fragments constituted of an swollen material after an impact stress of $0.6 \times \text{HEL}$,
- small fragments for glass-ceramic B after $0.2 \times \text{HEL}$, and swollen fragments after $0.6 \times \text{HEL}$.

Note the analogy with the features of samples recovered after spallation experiments.

For the $0.2 \times \text{HEL}$ level, we think that the samples were damaged by the second shock in the recovery box, since spallation experiments have shown that the material still had a non-zero spall strength after this impact stress. For the $0.6 \times \text{HEL}$ level, the swollen fragments recovered can be fingernail scratched. They have, however, a mechanical resistance which makes them very difficult to break up by hand. Certain fragments show large surfaces (0.5 to 1 cm^2) identifiable as part of the free surface of the target. Their thickness exceed 8 mm , a value to compare with the initial 5 mm thickness ! The presence of plane facets of considerable size and showing the layer of paint applied into the impact face before the test indicates that the dilatation occurs in the direction of the shock propagation. Furthermore, for the $0.6 \times \text{HEL}$ level, X-ray diffraction analysis indicates microplastic deformations of the crystalline phase.

Although many questions remain unanswered on the micromechanical aspects of shock-wave behaviour of glass-ceramics, the following propositions may be made. Plastic deformation of crystallites initiated from a threshold that is lower than the HEL (and even $< 0.6 \times \text{HEL}$) leads to a nucleation of microcracks at the interfaces between these crystallites and the glassy phase. This phenomenon has the effect of a spall strength which drops after an impact stress that is lower than the HEL. Howe-

ver, these microcracks are not interconnected at these stress levels, and the sample recovered at $0.6 \times \text{HEL}$ has the appearance of swollen material already described. The HEL at 8 GPa can be seen as an interconnection threshold for microcracks. The value of this limit, which is equal to the permanent densification threshold of fused silica, leads us to assume that this last phenomenon and the coalescence of microcracks are coupling. Comparison between results obtained on glass-ceramics A and B shows that the size of the crystallites has no significant modifying effect.

4 - BEHAVIOUR OF SINTERED ALUMINA

It is proposed by several researchers /24,25,26/ that the Hugoniot elastic limit of alumina corresponds to a state of a general microcracking. This proposition is supported by the lack of spall strength measured after an impact stress higher than the HEL. However, the results of the studies we have recently made on several aluminas (Table II) raise certain doubts as to its validity. Our experiments were of the type as those described for glass-ceramics. In addition, for aluminas, we measured spall strength and recovered samples after impact stress higher than the HEL.

Table II - Characteristics of sintered aluminas.

Material	Density	Percentage of alumina	Grain size (μm)	Supplying
A 16-2	3.91	≥ 99.7	4.7	E.N.S.C.I.*
A 16-11	3.54	≥ 99.7	1	id.
A 16-17	3.31	≥ 99.7	0.6	id.
A T -60	3.85	≥ 99.7	5 to 125	id.
UL 500	3.62	94	11	WADE (G.B.)

* Ecole Nationale Supérieure de Céramique Industrielle - Limoges - France.

4.1. - Results of spallation experiments

On all five types of alumina we always obtained non-zero spall strength even for loading up to twice the HEL. Taking experimental error into account, the spall strength is equal to the spall threshold at all stress levels (Figure 4). This spall strength is influenced by the porosity and the grain size but does not seem to be influenced by the purity. The result of plate-impact recovery experiments only relate to aluminas type A 16-2, A 16-17 and UL 500. The samples recovered after the spall tests show one single plane of rupture in the middle of the target.

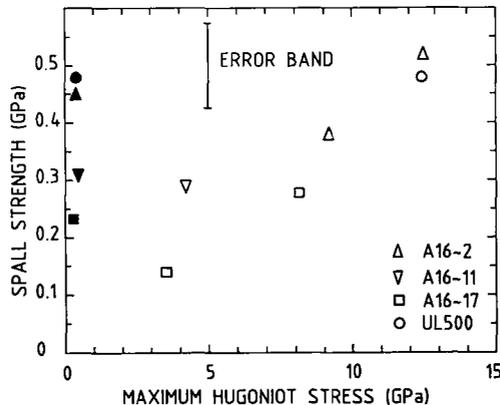


Fig. 4 - Spall strength versus maximum Hugoniot stress (or impact stress) for several sintered aluminas (Longy, 1987).

4.2. - Shock-wave compression : results and discussion

Characterizations of fragments recovered in the course of our study /23,27/ show the microplastic nature of shock-wave deformation of sintered alumina. This deformation occurs by generation and movement of dislocations in pure alumina. We have noticed very few twins and only at very high shock levels. X-ray diffraction analysis also points to the microplastic nature of the deformation of impure alumina UL 500. In this case, however, deformation is accompanied by damage revealed by dye penetration. SEM observation shows microcracks in the glassy intergranular phase. At the shock level we used, these microcracks are not interconnected ; the material remains coherent and there is no diminution in spall strength (Figure 4). We now discuss in detail the phenomenon of microcracking, then that of microplasticity.

4.2.1. - Phenomenon of microcracking

Microscopic examination shows no relationship between the HEL and microcracking. For pure aluminas, no microcrack was detected after an impact stress equal to $2 \times \text{HEL}$. If a few microcracks are detected in the impure alumina UL 500, the similarity of the velocity records /27/ in this and in pure alumina leads us to think that they do not have a rôle in the two-wave formation. Furthermore, if these microcracks played a rôle, we should find ourselves in the situation proposed by Lankford /25/, and the HEL would depend strongly on the strain-rate, through the crack inertia mechanism proposed by Grady and Kipp /4/. On an alumina similar to UL 500, Cagnoux and Longy /28/ have shown that the HEL remains invariant in relation to the strain-rate between 5.10^4s^{-1} and 6.10^5s^{-1} ; this confirms that the nucleation of a few microcracks has no influence on the formation of the two-wave structure.

We observe that the essential controlling factor of the appearance of microcracks is the presence of a glassy phase (which we have called unpurity). The two aluminas UL 500 /27/ and AD 85 /26/ which have the same porosity but a different proportion of glassy phase, have a same HEL value. This fact validates one discussion. We put forward the hypothesis that these microcracks are nucleated during unloading by tensile stress resulting from the heterogeneity of permanent deformation between the alumina grains and the intergranular glassy phase. We did not make recovery experiments on alumina T 60, so we are unable to come to a conclusion on the influence of the grain size on the microcracking phenomenon.

4.2.2. - Phenomenon of microplasticity

For all the aluminas tested, the HEL is superior to the grain microplasticity threshold. This plasticity is the only phenomenon with which the HEL can be linked. We are therefore led to suppose that the HEL represents a threshold from which the coupling between plastic deformation of grains and the macroscopic behaviour of the sample becomes effective. However, TEM observations revealed numerous grains which were not strained at $2 \times \text{HEL}$. In this phenomenon of grain microplasticity, the sample porosity has a modifying effect on the local stresses applied to each grain. For an identical applied loading, the higher the porosity the higher the local stresses applied to the grain. Thus the more porous is the sample, the lower is the plasticity threshold and the Hugoniot elastic limit. As for the grain size, it can be proposed that the bigger this is, the bigger are the initial microstresses which add to the loading to lower the plasticity threshold and therefore the HEL : The yield strength of the large grained alumina T 60 is much lower than that of a fine grained alumina with equivalent porosity (alumina A 16-2 for example).

5 - CONCLUSION

In conclusion, we resume the essential points about the shock-wave behaviour of the ceramics studied :

- Silica glasses

The elastic response of silica glasses is non-linear, which gives rise to the formation of a ramp precursor. The Hugoniot elastic limit is a permanent densification threshold for the silica. The evolution of this irreversible phenomenon produces

heat which softens the material. The consequences of this softening are to reduce the rigidity and the spall strength above the HEL.

- Glass-ceramics

The elastic response of the two-glass-ceramics studied is non-linear, which gives rise to the formation of a ramp precursor. However, this property cannot be linked to that of glassy matrix. The Hugoniot elastic limit seems to be a microcracks coalescence threshold. The permanent densification of the glassy matrix can play a part in this process. The nucleation of the microcracks before the HEL is certainly due to a plastic deformation of the crystallites. The two glass-ceramics tested lose their spall strength above their HEL, which is a consequence of their microcracking.

- Aluminas

The elastic response of aluminas is quasi-linear. The Hugoniot elastic limit is due to the microplasticity of alumina grains. This plastic deformation occurs by generation and movement of dislocations in pure alumina. In the case of aluminas with an intergranular glassy phase, the overall deformation of the sample is accompanied by a microcracking of the glass. The microcracks are not always interconnected at a stress level equal to $2 \times$ HEL for an alumina with about 10 % glassy phase ; this has the effect of creating a spall strength equal to the spall threshold of the initial material.

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