THE USE OF DIGITAL IMAGE CROSS-CORRELATION (DICC) TO STUDY THE MECHANICAL PROPERTIES OF A POLYMER BONDED EXPLOSIVE (PBX)

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The PBX studied is a polymer-bonded explosive that is formed from HMX and a nitrocellulose-based gelatinous binder. The physical and chemical properties of the PBX have precluded the use of existing whole-field optical techniques for the study of mechanical properties at the microstructural level. However, DICC, a non-contacting measurement technique, has been applied to the PBX with success. Virgin and thermally aged samples have been analysed to examine the change in mechanical properties of the PBX with time. It is found that both the stress and strain to failure of the PBX are decreased with thermal ageing. It is deduced that this degradation in mechanical properties is linked both to a reduction in the molecular mass of the nitrocellulose contained within the PBX binder and to a lesser extent other as yet unidentified mechanisms.

INTRODUCTION

Many methods exist to quantify the deformation of bodies under stress. Strain gauges and extensometers are commonly used to provide a measure of average displacement but they are unable to provide data on microstructural deformation. Several high-resolution non-contact optical techniques exist that are more sutable[1] and these have previously been applied to PBXs[2-4]. The PBX of interest is formed from HMX and ~1% nitrocellulose (NC) which is swollen with an organic plasticiser to form a gelatinous binder. The inherent mobility of the plasticiser has precluded the adhering of a phase-grating to the PBX surface, this being required for the use of optical Moiré interferometry, as used with other PBX compositions.[4-6] This paper reports the successful application of Digital Image Cross Correlation, a non-contacting imaging technique, to the study of the PBX microstructure and reviews the results from Brazilian mechanical testing.

DIGITAL IMAGE CROSS-CORRELATION

This technique operates through the mathematical comparison of two sub-images from a larger pair of displaced images, and determines the average motion between the two. If the process of comparing sub-images is repeated across the total image area, a full deformation map can be constructed. An illustration of the process is shown in figure 1.

It is important that the imaged surface is sufficiently random, such that the mathematical analysis algorithm identifies correlations with a clear single peak. Were a regular or grid-like surface analysed in this way, periodic displacements in apparently random directions would be found depending on the largest correlation peak found. Typically if the sample of interest is not sufficiently random, a random pattern is manually applied to the sample surface. For a PBX, in which a crystalline explosive is held within a binder matrix, a random surface pattern is revealed by careful sample polishing.

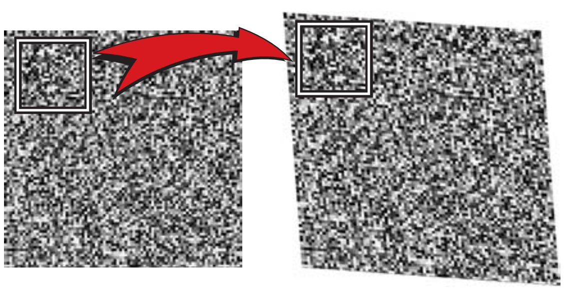

FIGURE 1. SCHEMATIC OF THE DICC PROCESS. MATHEMATICAL COMPARISON OF THE ORIGINAL SUB-IMAGE (LEFT) WITH THE DEFORMED ONE (RIGHT) DETERMINES THE AVERAGE MOTION.

The experiment commences by recording an image of the sample surface prior to the application of a displacement (e.g. *via* the Brazilian mechanical test). In most cases, the displacement is caused by the application of stress to the imaged object. Repeated images are recorded at some subsequent increases in stress, thus building a sequence that spans the entire test. The images may be recorded onto film for later digitisation, or directly recorded onto a CCD (charge-coupled device) chip within a camera.

Two methods can be used to analyse the captured images. The first method is to compare each incremental frame with the initial (undeformed) frame. The second method compares an image only with the one that immediately precedes it. The former method results in lower errors, provided that the total displacement between the first and the last image is sufficiently small that the correlation algorithm can find a crisp peak. For the reported experiments, the deformations were small enough to enable both analysis methods to be performed, with generally excellent agreement.

The algorithm used for the analysis was written by Sjödahl[7-9]. This method performs the correlation in the frequency domain using Fourier transforms, rather than the spatial domain. The Fourier approach offers a considerable computational speed advantage, allowing multiple correlation iterations to be performed, therefore increasing the resulting displacement accuracy. With a given pair of optimal images, the algorithm is accurate to between 0.01 and 0.05 pixels. For the microstructural images generated from a PBX, previous experiments have shown that the algorithm is accurate to approximately 0.1 pixels[10].

In order to obtain the average strain over the image (in addition to the shear strains if desired) a two-dimensional plane is fitted to the displacement data. In this way, any rigid body motion between the images can be removed. Alternatively, if a graphic representation of the motion is required, it can be achieved using displacement vectors overlaid upon the source image. An example of the vector output from a tested sample of the PBX is illustrated in figure 2.

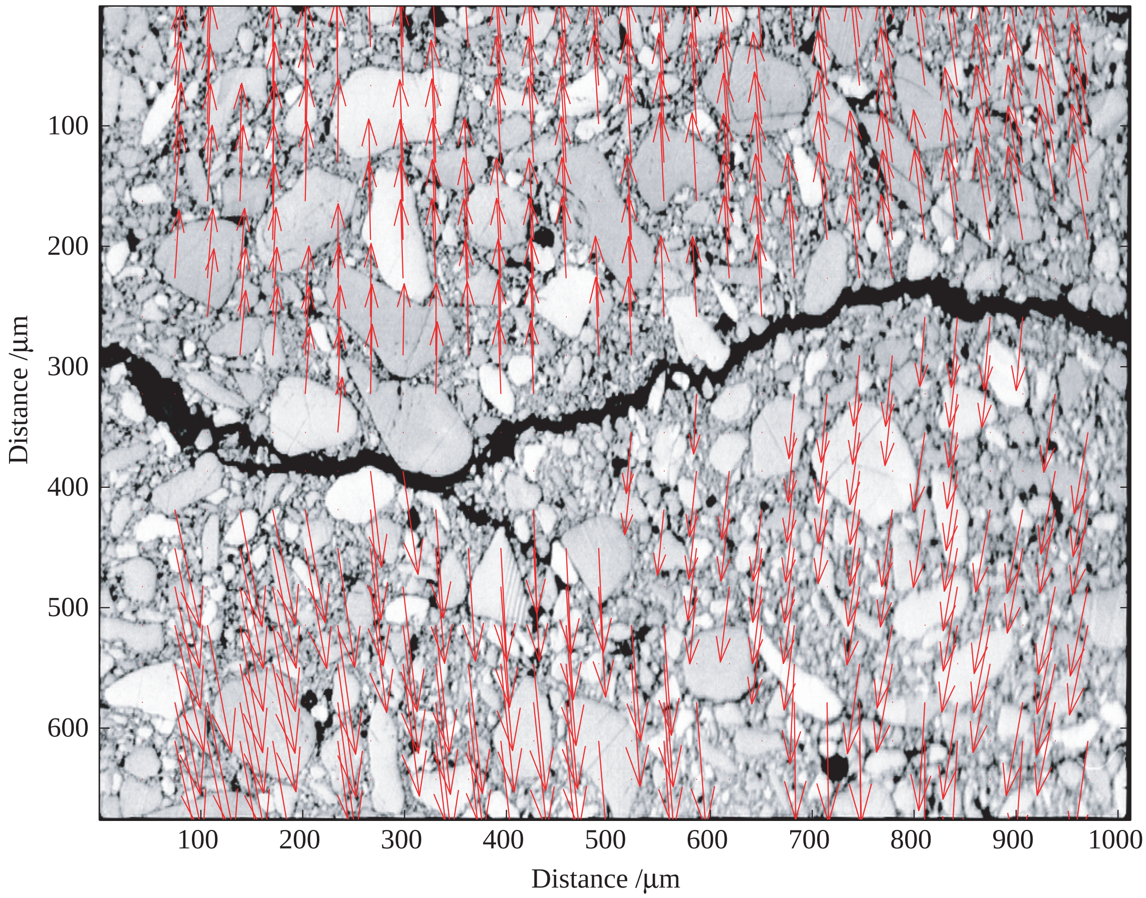

FIGURE 2: EDC 37 LOADED TO FAILURE DURING A DICC TEST. THE VECTORS INDICATE THE AVERAGE DISPLACEMENT MEASURED AT EACH SUB-IMAGE.

THE BRAZILIAN TEST

The safety requirements of the test laboratory preclude the use of standard uniaxial dumbbell testing on multi-gram quantities of a PBX. However, other test methods exist which can be applied to samples that contain less than 0.5 g of explosive. The Brazilian, or diametral compression test, is a biaxial method for determining the tensile failure stress of a material *via* the application of a diametric compressive load to a cylindrical sample. The test geometry is illustrated in figure 3.

Simple elastic theory[11] predicts that the tensile stress at the centre of the test sample is given by

$$\sigma_x = \frac{2P}{\pi D t}, \qquad (1)$$

where P is the applied force, D is the sample diameter and t is the sample thickness. The compressive stress at this point is greater than the tensile stress by a factor of three. If the full elastic expressions are evaluated over the entire area of the disc, it is found that the tensile stress along the vertical diameter remains comparatively uniform at the disc centre, thus validating the test. It has been shown that curved anvils tend to lower the shear stress at the anvil, which in turn prevents premature sample failure[12]. The effect upon the central stress has also been reported in the same paper, with the derivation

$$\sigma_x \approx \frac{2P}{\pi D t}\left(1-\left(\frac{b}{R}\right)^2\right), \qquad (2)$$

where b is the contact half-width of the anvils and R is the radius of the specimen. The curvature of the anvils can be chosen as desired, down to a minimum b/R ratio of 0.27, which represents the limit for purely tensile failure. In the reported experiments, a b/R ratio of 0.30 is assumed, based upon previous testing of PBX materials. Substitution of this value into (2) gives a correction factor of 0.91 upon the stress predicted by (1).

TABLE 1. SAMPLE AGEING DATA.

Sample Name	Ageing Temp. / ˚C	Ageing Time / days	Molecular Weight Mn /Mw / 10^3
20/550/C	20	550	124/497
20/550/W	20	550	†
40/4380/C	40	4380	†
46/150/C	46	150	133/456
46/300/C	46	300	136/456
56/150/C	56	150	117/389
56/300/C	56	300	92.6/343
66/150/C	66	150	67.8/187
66/300/C	66	300	39.2/119
66/666/C	66	666	30.2/52.5
LMW/W	20	365	123/315
46/300/W	46	300	†
56/300/W	56	300	†
66/300/W	66	300	48.3/121

† Data not yet available

EXPERIMENTS

Samples of the PBX, nominally 10 mm in diameter and 4 mm thick, were machined at AWE. The nitrocellulose in the PBX originated from two cellulose feedstocks: cotton linters and wood pulp. In addition to the virgin (unaged) explosive, samples were supplied that had been aged at elevated temperatures for up to ~4400 days A sample from an identical composition manufactured with a lower molecular weight nitrocellulose than standard was also supplied. The ageing conditions of the samples are reproduced in table 1.

The time at temperature indicated for the ambient control samples (20/550/C and 20/550/W) indicate that the samples were stored at 20±4 °C for approximately 550 days prior to testing. The low molecular weight material (LMW/C) was stored at ambient temperature for approximately 365 days prior to testing. Additionally the thermally aged samples were also stored at 20±4 °C for the same duration. Separate studies have indicated no measurable effect upon the mechanical properties or nitrocellulose molecular weight from prolonged storage (over periods of years) at temperatures less than 25 °C. The molecular mass of the nitrocellulose extracted

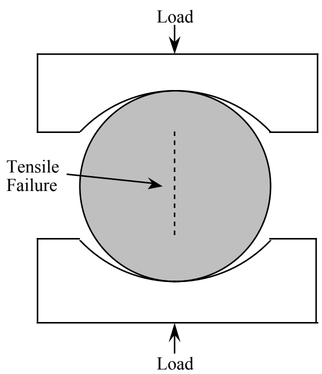

FIGURE 3. THE BRAZILIAN TEST GEOMETRY.

from the PBX samples after processing is also reported in table 1.

It should be noted that this data was determined using a gel permeation chromatography (GPC) technique which generates molecular weight moments that are relative to polystyrene standards.

The manufactured samples required polishing for the DICC experiments. It is conceivable that this process may induce damage or otherwise change the properties of the sample, hence a number of samples were deformed in the Brazilian apparatus without polishing in order to establish a baseline failure stress. The average diametral strain to failure was also measured, using a modified extensometer. In the case of the DICC experiments, only the strain at the centre of the Brazilian sample, ~1 mm^2, was measured. For comparison, it has been shown that if a PBX is assumed to be a perfect isotropic solid, the measured centre strain should be greater than the average diametral strain by a factor of approximately 2.1.[14]

RESULTS

The average diametral strain, central strains to failure, as-received and polished failure stresses of the tested samples are presented in table 2.

It is observed that the results from the virgin cotton linter and wood based materials (20/550/C and 20/550/W) are very similar, suggesting that the biological source of nitrocellulose does not influence the initial PBX mechanical properties. It appears that the polishing process causes an approximate 10% reduction in the failure stress for the unaged PBX, however this factor is found to be 20% for the thermally aged material.

Broad trends can be observed from the ageing data. The stress and strains to failure of the unpolished samples appear to decrease with increased ageing duration and temperature. The polished (DICC) samples also exhibit similar broad trends.

There are anomalies within the data: it would appear that the material aged for 300 days at 46 °C is slightly stronger than material aged for only 150 days at the same temperature (it is interesting to note that the determined molecular weight of the nitrocellulose within the two samples is comparable.) In addition, the diametral strain to failure of the material aged at 56 °C for 150 days appears to be greater than for the material aged at 46 °C for the same duration.

It has been deduced that the predominant mechanism for nitrocellulose degradation under mild ageing conditions is scission of the acetal linkage within the polymer chain,[15] which results in a decrease in the molecular weight of the polymer, and would impact upon the mechanical properties of a nitrocellulose-containing PBX. It is observed that the molecular mass of the tested low molecular weight formulation is similar to that of standard material that has been aged for between 150 and 300 days at 56 °C. The stress and strain values of this low MW material are also comparable to the aged materials, reinforcing the relationship between NC molecular weight and PBX mechanical properties.

CONCLUSIONS

Digital Image Cross Correlation, in tandem with the Brazilian mechanical test, has proven effective for the investigation of microstructural strain in the PBX. DICC can generate useful central strain data for samples undergoing a mechanical deformation, however the required polishing technique reduces the stress to failure of the unaged PBX by 10% and by 20% for thermally aged material.

Examination of unaged material with wood and cotton linter nitrocellulose feedstocks suggests no variation in initial material mechanical properties.

The ageing of the PBX at elevated temperature results in a decrease in the molecular mass of the nitrocellulose component. This result is mirrored in a reduction in the stress and strain to failure of the material.

TABLE 2: STRESSES AND STRAINS TO FAILURE IN THE PBX

Sample Name	Diametral Strain to Failure / millistrain	DICC Strain to Failure / millistrain	As Received Failure Stress / MPa	Polished Failure Stress / MPa
20/550/C	2.6±0.4	3.89±1.13	0.98±0.06	0.85±0.13
20/550/W	2.3±0.2	†	1.03±0.06	†
40/4380/C	1.2±0.2	3.3±0.3	0.60±0.1	0.55±0.05
46/150/C	1.1±0.1	3.62±1.0	0.89±0.07	0.71±0.06
46/300/C	0.88±0.07	2.6±0.8	1.07±0.04	0.81±0.08
56/150/C	1.45±0.45	3.1±1.2	0.94±0.03	0.76±0.08
56/300/C	0.64±0.19	2.9±0.6	0.77±0.13	0.65±0.10
66/150/C	1.20±0.6	3.6±0.9	0.49±0.13	0.44±0.05
66/300/C	<0.2	2.1±0.5	0.33±0.05	0.30±0.04
66/666/C	<0.2	2.2±0.1	0.35±0.14	0.20±0.01
LMW/W	0.86±0.03	3.8±0.6	0.74±0.02	0.70±0.04
46/300/W	0.81	3.0±0.6	0.86	0.69±0.08
56/300/W	0.84	†	0.68	†
66/300/W	0.89±0.01	3.5±0.9	0.47±0.06	0.36±0.02

† Data not yet available

The mechanical properties of the low molecular weight PBX formulation broadly equates with aged (standard) material of similar nitrocellulose molecular weight. However, the variability between samples of the unaged, but lower molecular mass PBX, is less than between samples that have been artificially aged to a similar molecular mass.

Thus while chain scission occurs during ageing, it appears that aged material cannot be totally mimicked by starting with lower molecular mass material. Therefore it is deduced that while molecular changes within the nitrocellulose polymer have a profound effect upon the resultant PBX mechanical properties other, as yet, unidentified factors also play a role.

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