



## New samples with artificial voids for ultrasonic investigation of material damage due to creep

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### ABSTRACT

This paper describes the application of acoustic birefringence measurement for evaluation of material degradation due to creep. The problem with ultrasonic investigation of the influence of creep-induced voids on ultrasonic wave velocities is the lack of samples containing well-defined voids. The only way to “produce” damaged material with numerous voids is accelerated creep laboratory test or long-term service of material in high temperature and stress. To describe voids it is necessary to perform laborious metallographic examination of damaged material. Paper presents glass samples with artificial semi-flat voids created with laser engraving technique. Results of ultrasonic wave velocities and acoustic birefringence measurements in glass samples proved that laser engraving can be helpful for the experimental investigation of voids influence on solid acoustic properties.

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### 1. Introduction

Steel subjected for a long period of time to stress and high temperature can be damaged due to creep. Most often the results of material degradation are small voids or cavities which emerge on grain boundaries and, in general, are oriented perpendicular to dominant stress direction. During creep progress, small voids usually coalesce and form micro cracks which decrease material strength and eventually can lead to material rupture. Detection and evaluation of material degradation due to creep is vital in many industries like power plants or chemical installations. Numerous destructive and non-destructive techniques have been developed to evaluate material degradation. They include metallographic investigation [1,2], magnetic properties evaluation [3] and ultrasonic testing.

In contrast to other techniques mentioned above, the ultrasonic methods have potential of detecting material changes not only near the surface but also in the bulk of material. Voids (cavities), although much smaller than the wavelength, affect ultrasonic pulse velocity and attenuation. Correlation of these acoustic parameters with creep induced damage was investigated for various steel grades. Usually measurements were performed on samples subjected to accelerated creep in a laboratory conditions and decrease of wave velocity and rise of attenuation was observed in course of creep. For example in [4] measurements of ultrasonic waves velocity in stainless steel undergoing creep

were presented. Some authors inform about acoustic anisotropy caused by creep-induced damage [5,6]. But precise measurements of ultrasonic velocity or attenuation in industrial conditions are very difficult if possible at all. To improve the precision of such measurements contactless electro-magnetic transducers (EMAT) were used to investigate correlation between attenuation and creep damage [7].

Only a few papers describe full-size ultrasonic investigation of creep. For example in [8] the authors describe ultrasonic examinations performed on a 3 m long pipe section subjected to a creep test. Precise measurements of velocities on such object, without destructive sample preparation, were impossible and to detect creep damage acoustic noise resulting from aggregates of creep voids was measured. Acoustic noise evaluation was expected to detect creep damage before cracks formed in the final stage of component life became big enough to be detected with a standard TOFD technique. The noise measurements were done in heat-affected zones of the pipe welds.

Difficulties in utilization of ultrasonic wave velocity and attenuation as a practical tool for material damage estimation are simple. To determine the absolute value of ultrasonic velocity it is necessary to measure the actual material thickness what is often unfeasible on the real installations (e.g., walls of pressure vessels and pipelines). It should also be convenient to know the velocity in the as delivered (non damaged) state of material what is quite often impossible. The same applies to attenuation coefficient that is even more prone to measurement errors than velocity measurements. The most common difficulties in this case are surface curvature, roughness and lack of parallelism. Also the influence of material damage on nonlinear acoustic parameters

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[9,10] seems to be extremely difficult to investigate in industrial conditions.

One of the promising ultrasonic techniques, based on the shear wave velocity changes, is a measurement of acoustic birefringence  $B$ . This parameter can be measured on flat objects like plate or pipe wall, with shear waves propagating in the thickness direction and polarized in two orthogonal directions which are acoustic axis of a material under investigation. Birefringence value is calculated from

$$B = \frac{(V_{12} - V_{13})}{(V_{12} + V_{13})/2} = \frac{(t_{13} - t_{12})}{(t_{13} + t_{12})/2} \quad (1)$$

where,  $V_{12}$ ,  $V_{13}$  is the velocities of shear wave propagating in the direction 1 and polarized in directions 2 and 3, respectively;  $t_{12}$ ,  $t_{13}$  are the times of flight of shear wave propagating in direction 1 and polarized in directions 2 and 3, respectively; 1 is the thickness direction; 2 is the direction perpendicular to max. tension stress; 3 is the maximum tension stress direction.

As can be seen from formula (1)  $B$  is determined without any information concerning material thickness. Acoustic birefringence is a measure of acoustic anisotropy and in steel components its value depends on several factors. They are material texture (preferred grain orientation) introduced during manufacturing, stress state which influence shear wave velocities through elastoacoustic phenomena and material damage due to fatigue or creep (oriented voids or micro cracks).

In investigation of material damage the birefringence technique has several advantages. It provides information about material properties averaged over the material thickness (in the bulk of material). As both polarization waves propagate along the same paths in the plate or pipe wall, the local variations of material thickness, composition or surface condition does not affect the measured birefringence value. Also the temperature does not influence the result of measurements, because it can be reasonably assumed that the velocity temperature dependence is the same for both polarizations of the shear wave.

In many industrial installations, like pipes for example, the directions of dominant stresses are known and the general orientation of expected voids which can emerge due to creep can be predicted. So the directions of shear wave polarization, to measure acoustic birefringence, can be assumed. All above makes acoustic birefringence measurements much easier to apply in industrial conditions than measurements of ultrasonic velocities or attenuation. These practical advantages of acoustic birefringence technique were practically proven in ultrasonic evaluation of residual stresses in railroad wheel rims. In this application, where precise measurements must be performed on used railroad wheels in workshops or in wagons on the track, the birefringence measurement has been in wide use for several years, for example [11–13].

Expectation that the acoustic birefringence measurements can be a useful tool in damage detection could be inferred from the earlier works. For example in [5,14] the changes of shear wave velocities in steel due to creep were measured and reported. However, values of the acoustic birefringence have not been calculated and the acoustic birefringence has not been considered as a separate diagnostic parameter for detection of material damage due to creep.

## 2. Measurements of the acoustic birefringence in steel samples

The acoustic birefringence was measured in steel samples made of several steel grades. Details of sample preparation, investigation of mechanical properties and ultrasonic measurements are described elsewhere [15]. Here, we present only results

obtained for the group of 8 specimens made of 40HNMA steel and subjected to accelerated creep test. This steel grade is commonly used for manufacturing elements working at elevated temperatures. Steel samples were subjected to uniaxial tension creep test at temperature 773 K and stress 250 MPa. In order to assess a damage development during the process of creep the tests were interrupted for a range of the selected time periods 100, 241, 360, 452, 550, 792, 929 and 988 h, which correspond to the increasing amounts of creep strain equal to 0.34%, 0.8%, 1%, 1.1%, 1.2%, 2.3%, 4.0%, and 6.5%, respectively. Rupture of the sample was observed after 1024 h. Creep curve of the 40HNMA with points representing interrupted creep tests is presented in Fig. 1.

After creep tests acoustic birefringence was measured in samples and calculated according to the formula (1) where  $V_{12}$  and  $V_{13}$  denote the shear waves propagating in the thickness direction and polarized respectively, perpendicular and parallel to the creep stress direction. Values of acoustic birefringence were evaluated in several points on the loaded (damaged) part of the specimen and in the non-deformed fixture parts.

The measurement results are illustrated in Fig. 2 in dependence on the total creep strain of the samples. The points designated  $B_{av}$  show average values of birefringence measured in the loaded parts of specimens. The average birefringence increases in course of deformation reaching a value of 0.84% for sample no. 8. The birefringence in the ruptured sample was not measured because of strong sample necking. For comparison the birefringence measured in fixture parts of the same specimens shows only small scatter around zero. This scatter can be considered as a result of initial material inhomogeneity and the measurement accuracy. Fixture readings show also that long-time exposition to high temperature does not influence the birefringence value. The point designated  $B_{max}$  shows maximum value of birefringence detected in the loaded parts of specimens. It can be seen that for smaller deformations (up to 4%) the maximum and average values do not differ considerably showing the uniformity of material state along the sample length. For higher deformations the creep damage distribution seems to be not uniform and specific locations on the loaded part of sample exhibit a considerably higher birefringence values. For the most deformed sample the maximum value of birefringence reached 1.5% and was about twice as large as the average value on the whole loaded part.

In samples subjected to accelerated creep test acoustic birefringence changes can be a result of two different physical

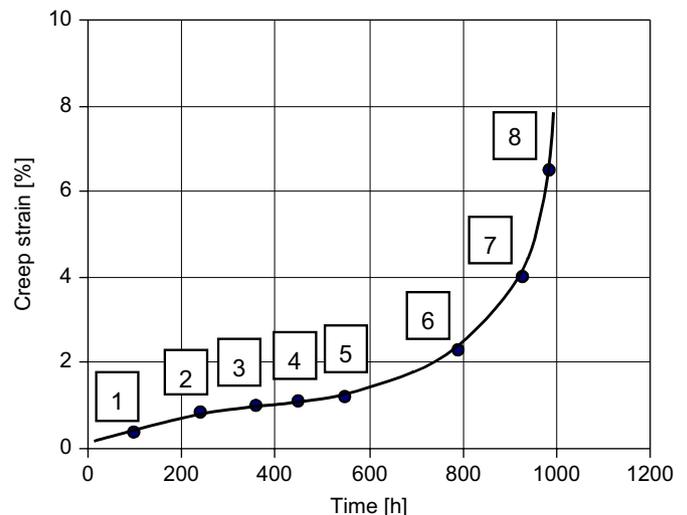


Fig. 1. Creep curve of the 40HNMA steel with points representing interrupted creep tests.

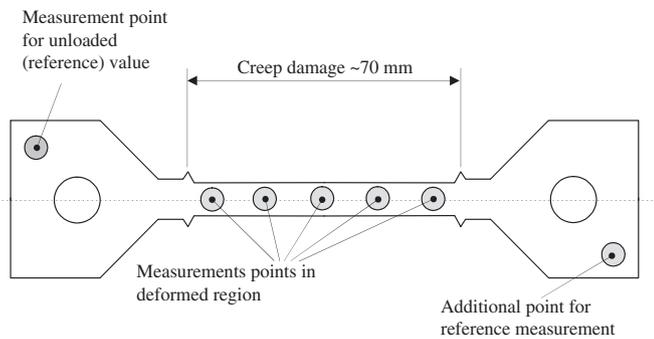


Fig. 2. Accelerated creep sample showing locations of ultrasonic measurements.

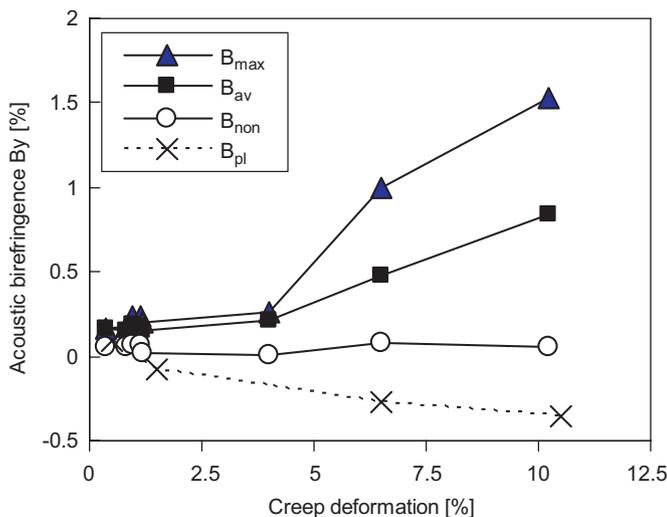


Fig. 3. Acoustic birefringence dependence on creep deformation in 40HMNA steel samples.  $B_{max}$ —maximum value of birefringence measured in the deformed area of the sample,  $B_{av}$ —average value of birefringence in the deformed region,  $B_{con}$ —average value of birefringence in non-deformed control (reference) regions,  $B_{pl}$ —maximum value of birefringence in samples after room temperature tension test.

phenomena: the plastic deformation leading to change in grain orientation distribution (texture), formation of oriented voids and micro cracks on the grain boundaries. The first mechanism leads to negative change in birefringence value, the second one—to positive change in the birefringence value. This interpretation was confirmed by the measurement of birefringence in samples of the same geometry and made of the same steel grade subjected to standard tension tests at room temperature. Results are presented in Fig. 3 showing the dependence of birefringence value on the plastic deformation of 40HMNA steel samples.

It can be seen that birefringence value in loaded parts of the samples drops down in course of plastic deformation reaching a value of  $-0.4\%$  in the most deformed sample. It is completely opposite dependence than in case of similar samples subjected to creep deformation. The apparent reason is the lack of oriented voids and micro crack which caused the increase of birefringence value in case of creep tested samples.

It is also worth to mention that the initial values of birefringence, measured before creep and tensile tests, was relatively small (below  $0.15\%$ ). It can be said that for steel grades under investigation initial state of material, in terms of birefringence value, is well defined. Every substantial deviation of birefringence value detected on working installation, depending on its sign, can be attributed to plastic deformation (texture

change) or creep damage (oriented voids and microcracks). Theoretically, in certain conditions the two concurring effects can cancel each other leaving birefringence value unaffected. In industrial practice however steel elements are subjected to long time and relatively low-stress creep conditions resulting in low deformation and creep cavities or voids formation.

The figures presented above show relation between the acoustic birefringence and sample deformation which is not a direct measure of its material degradation. In other papers acoustic parameters of sample subjected to creep test are correlated with density or porosity of damaged material [16]. Density changes are evidence of cavities and inform about total volume of voids but not about their number, sizes and orientation. To obtain detail information concerning voids caused by creep, the only method is a laborious destructive metallographic examination of material samples. This procedure relies on removing a thin material layer, preparing the material surface, detection and registration of opened voids, and requires repeating this process layer by layer. Therefore, it is very difficult to compare ultrasonic readings with material damage defined in terms of voids density, sizes and orientation. It is also very difficult to “manufacture” steel samples with known number and sizes of cavities. Samples of damaged material, containing voids due to creep, can be taken from service exposed components, after many years of operation. This way of “producing” samples is, however, destructive and the only information concerning damage, which can be easily measured, is material density. Therefore, the results of experimental ultrasonic investigations are usually compared to creep-induced strain or material density. Lack of detailed information concerning voids in the samples makes difficult not only to find correlation between the acoustic parameters and the actual material damage but also makes difficult to verify the theoretical models describing the velocities of ultrasonic waves propagating in damaged solid, which contain numerous small and well-defined cavities.

### 3. Measurements of the acoustic birefringence in glass samples

The well-known application of ultrasonic technique is non-destructive flaws detection. Ultrasonic devices are usually calibrated on calibration block containing various reflectors of ultrasonic waves. Such reflectors, called “artificial flaws”, are made as flat bottom holes of various diameters, side holes or groves of various depths. In the block made of steel, such simple geometry reflectors are relatively easy to manufacture.

For ultrasonic investigation of material damage, it would be convenient to have the reference blocks containing artificial voids, imitating material damage due to creep. Such blocks, with various void sizes, orientations and densities, could be used for birefringence method calibration. Unfortunately manufacturing of such block made of steel nowadays seems to be impossible.

One of the few technique which is able to produce quickly and in a controlled way small voids in a solid is a sub-surface laser engraving (SSLE). The generation of small, visible dots or pixels inside glass lenses, used in high-power laser technology, was originally a problem known as “Laser Induced Damage”. Although the laser produced a powerful beam of light which affected glass structure in the beam focus, the surface of the lens was not damaged. It was discovered that the glass damage was a result of the phenomena which is known today as Multi-photon Absorption [17] and nowadays is used for laser 3D engraving to produce scenes, logo or “portrait sculptures” in blocks of glass. Machines used for subsurface engraving are equipped with water-cooled, solid-state neodymium:yttrium aluminum garnet (Nd:YAG) lasers producing pulses of 532 nm wavelength light, operating in a pulse

mode. Laser pulse energy ranges from 2.0 to 60MJ and pulse duration from  $10^{-11}$  to  $10^{-8}$ s. Usually each “pixel” generated inside the glass has a form of an elongated “branch” of micro cracks. Depending on laser pulse parameters dimensions of the individual “pixel” (diameter/length) vary from 0.07/0.1 to 0.3/0.5 mm and spacing between pixels can be as close as 0.005 mm [18].

Computer-controlled focused laser beam can precisely engrave numerous “pixels” to form easily visible white surfaces inside the glass block. Transparency of such surface, necessary to obtain 3D impression, depends on spacing between pixels. To obtain good-looking 3D image pixels are usually spaced no closer than 0.15 mm. Tighter spacing of pixels can result in crack formation damaging the glass between adjacent pixels and ruin the picture. In terms of 3D portrait engravings such cracks are an unwanted flaws, disturbing the image. However, from the point of view of ultrasonic testing of materials such cracks are small, almost flat discontinuities modifying acoustic properties of a solid. Controlled action of a laser beam is able to create cracks of various size, shape and orientation in a volume of glass block. Small cracks, spread randomly in the glass volume can form a sample for ultrasonic investigation of material damage.

In the initial state the glass used for 3D engraving is an isotropic material with velocity of longitudinal and shear waves equal to 5590 and 3360 m/s, respectively; and very low attenuation. Comparing the acoustic properties of steel, the longitudinal wave velocity in such a glass is only about 5% lower and the shear velocity—about 4% higher. To investigate the influence of laser-produced cracks on acoustic properties of glass, cubic samples with multiple, randomly distributed, semi-flat micro cracks were produced by laser 3D engraving. Sample size was  $30 \times 30 \times 30 \text{ mm}^3$ . To avoid sample surface damage cracks form a  $20 \times 20 \times 20 \text{ mm}^3$  inside the sample. Sizes of the individual pixels generated in the glass samples are about 0.1 mm in diameter and about 0.3 mm long. To create semi-flat crack several laser actions were used with focal point spacing about 0.1 mm. To test the influence of the laser induced damage on acoustic properties samples with various densities of cracks were prepared. In all samples cracks were distributed randomly in the glass volume and were oriented parallel to one side of the cube sample. Table 1 lists the number of laser actions, crack sizes and crack densities in the glass samples.

Fig. 4 presents three samples with various densities of cracks ranging from 200 up to 1000 cracks in  $1 \text{ cm}^3$ . The average crack size was determined by the number of laser beam actions forming one crack. For the samples presented in Fig. 3 the average crack size evaluated under optical microscope was about 0.53 mm and all cracks were oriented (more or less) perpendicular to the sample x-axis. All elongated “branches” forming cracks in the glass were oriented along the z-axis, i.e. along the direction of the laser beam. For crack density  $1000/\text{cm}^3$  two samples were prepared with different average crack size 0.53 and 0.88 mm, respectively. Fig. 5 presents schematically the shapes of several micro cracks in the sample A (4 laser actions/crack) as seen from different directions. Semi-flat shape can be seen looking along x-axis and branches of almost separated small, very thin, flat crack

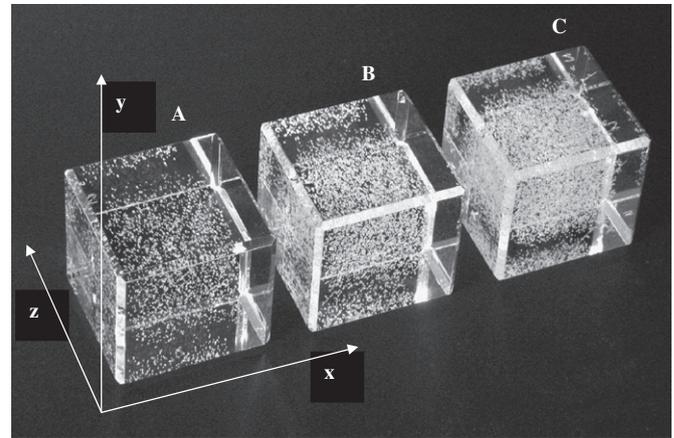


Fig. 4. Three glass samples with laser-induced multiple microcracks. Sample A has a crack density of  $200/\text{cm}^3$ , sample B of  $500/\text{cm}^3$  and sample C of  $1000/\text{cm}^3$ .

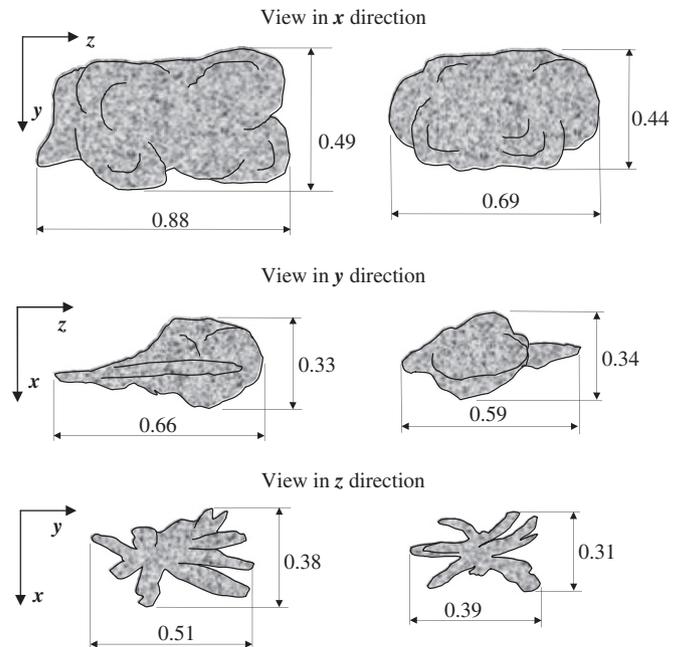


Fig. 5. Schematic shapes of several micro cracks in the glass sample A, each formed by 4 laser interactions, seen from different directions (dimensions in mm).

looking along z-axis. Cross-section of each micro crack is the biggest in x- and smallest in the z-direction.

Micro cracks produced in the glass are typical for laser engraving technique and are not identical with voids which develop in steel, in the late creep stages. However, they are small, semi-flat and rough surface discontinuities, which seems to be a better approximation of natural voids in crept metal than ideal penny-shape, or flat oblate spheroids used in theoretical modeling.

The smallest laser produced micro cracks are about 0.5 mm long and about 0.35 mm high and are bigger than voids in metals in the early creep stages. In the later creep stages, however, sizes of voids in the steel can reach fraction of mm [4,19]. It means that laser-induced artificial cracks can imitate material damage due to creep at least in the late stages of creep process.

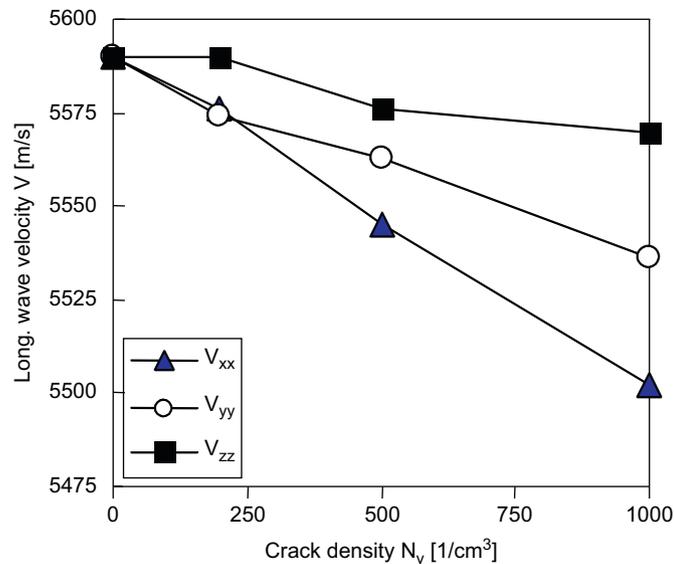
The glass samples shown in Fig. 4 seems to be fully filled in with white cracks. However, these small cracks are not a barrier for ultrasonic waves, which can easily propagate in all directions

Table 1  
Crack densities and average sizes in the glass samples.

Sample Nr	No. of laser interactions forming 1 crack	Crack density ( $\text{cm}^{-3}$ )	Average crack size (mm)
A	4	200	0.53
B	4	500	0.53
C	4	1000	0.53
D	8	1000	0.88

**Table 2**  
Velocities of ultrasonic waves propagating in damaged volumes of glass samples.

Sample	Longitudinal waves			Shear waves	
	$V_{xx}$ (m/s)	$V_{yy}$ (m/s)	$V_{zz}$ (m/s)	$V_{yx}$ (m/s)	$V_{yz}$ (m/s)
Virgin	5590	5590	5590	3365	3365
A	5576	5574	5590	3359	3363
B	5545	5563	5576	3346	3357
C	5502	5536	5570	3332	3350
D	5280	5460	5566	3290	3357



**Fig. 6.** Relation of longitudinal wave velocities to the density of laser-induced cracks in glass samples.

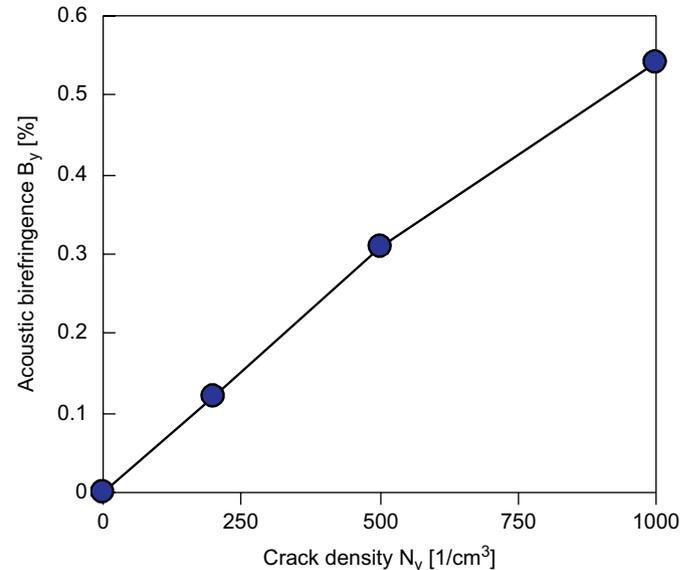
in the samples. Even for sample D, containing about 1000 cracks in each cubic centimeter, the acoustic noise level was difficult to measure with 2.25 MHz ultrasonic longitudinal wave. The acoustic properties of the samples were measured with the longitudinal and shear waves, both at 2.25 MHz frequency. For this particular frequency the wavelengths are bigger than the crack sizes and equal, respectively, to 2.5 mm for the longitudinal and 1.5 mm for shear waves.

Table 2 presents the velocities of the longitudinal and shear waves measured for waves propagating in damaged area along three samples axis. Velocity  $V_{ij}$  denotes velocity of the wave propagating in  $i$ -direction and polarized in  $j$ -direction. In accordance with expectations, the velocities of ultrasonic waves in damaged samples are lower compared to the virgin sample. The most significant velocity drop is observed for longitudinal waves traveling perpendicularly to cracks orientation ( $V_{xx}$ ). The velocities of longitudinal waves propagating in other directions drop less. The dependence of the longitudinal velocities on the cracks density is shown in Fig. 6.

It can be seen that all velocities drop with the crack density in a linear manner. The difference between  $V_{yy}$  and  $V_{zz}$  can be explained by the crack morphology induced by the laser generation method. Anisotropy of material in plane parallel to cracks is the result of a specific shape of each individual laser “pixel” (see Fig. 5). As it was mentioned earlier each laser pulse produces elongated “branch” rather than a dot. Any sample made with laser beam operating from one direction, with all “branches”

**Table 3**  
Acoustic anisotropy measurements of damaged glass samples.

Sample	Virgin	A	B	C	D
Birefringence of shear waves $B_y$ (%)	0	0.12	0.31	0.54	2.0
Relative difference of longitudinal wave velocities $A_{xy}$ (%)	0	0.25	0.56	1.22	5.0



**Fig. 7.** Birefringence of shear waves propagating in the  $y$ -direction as a function of crack density.

oriented parallel to one axis, is by its nature anisotropic. These phenomena were observed also on samples with bigger laser “pixels” spacing without any linking cracks between adjacent pixels. Shear wave velocities were measured for wave propagating in  $y$ -direction and polarized in  $x$ - and  $z$ -directions. For both wave polarizations velocity decreases with increase of crack density. However, velocity changes for the wave polarized in  $z$ -direction are much smaller. Corresponding birefringence of shear waves and anisotropy factor for longitudinal waves calculated from the velocity measurements, are presented in Table 3. Acoustic birefringence was calculated as  $2(V_{yz}-V_{yx})/(V_{yz}+V_{yx})$  and longitudinal waves anisotropy factor  $A_{xy}$  as  $2(V_{xx}-V_{yy})/(V_{xx}+V_{yy})$ .

From the data shown in Table 3, it is clear that anisotropy factor for longitudinal wave velocity  $A_{xy}$  is considerably greater than birefringence  $B_y$  of shear waves. Unfortunately this type of parameter can be precisely measured only on cubic-like samples in laboratory conditions. In practical applications on pipes or pressure vessels walls the accurate measurement of difference of longitudinal velocities in two perpendicular directions is much more problematic than measurement of shear wave birefringence.

The graphical representation of shear wave birefringence dependence on crack density in glass samples is illustrated in Fig. 7. Again it can be seen that birefringence value is almost linearly depended on crack density for a given crack size. The maximum birefringence value for the series of samples with average cracks size of 0.53 mm was about 0.54%. The dependence of the birefringence value on the average crack size is depicted in

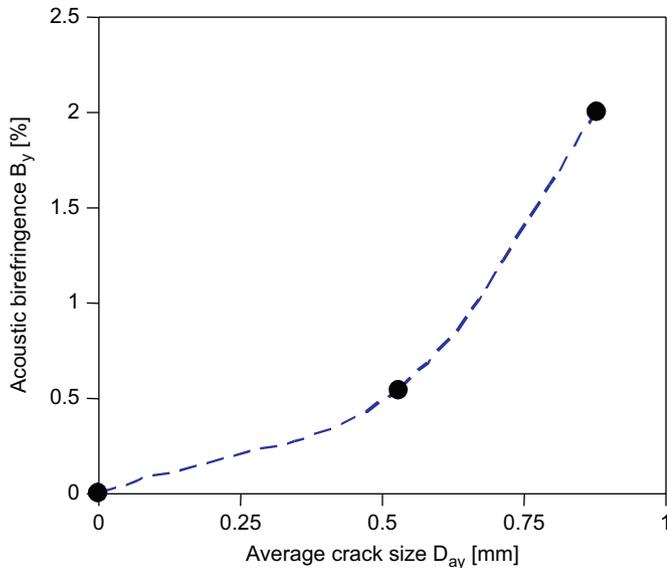


Fig. 8. Birefringence of shear waves propagating in  $y$ -direction as a function of average crack size for a constant crack density  $1000/\text{cm}^3$ .

Fig. 7. The birefringence increases rapidly with average crack size. As can be seen in Fig. 8, where the trend line was also drawn, the functional dependence is near to  $D^3$ .

The results of the ultrasonic measurements presented above show that laser engraved, small parallel-oriented micro cracks made glass sample anisotropic. The birefringence changes found in the glass samples with controlled micro crack density confirm the general interpretation of ultrasonic results obtained on the steel samples subjected to creep tests. The gathered numerical data also give rough estimation of the density of oriented micro cracks needed to produce a given value of ultrasonic birefringence.

#### 4. Conclusions

Creep-induced voids in the material influence ultrasonic wave velocity. The wave velocities depend not only on voids density and size but also on voids orientation in relation to the wave propagation direction. For shear waves velocity depends also on wave polarization. The acoustic birefringence resulting from this dependence seems to be a good parameter to monitor material degradation due to creep.

The advantage of birefringence measurement, comparing to velocity or attenuation measurements, stems from its simplicity and applicability in industrial conditions. The obvious restriction is that it can be effectively applied only at the late stages of creep when numerous oriented voids and micro cracks are formed.

To investigate the influence of voids on ultrasonic waves velocities and to verify theoretical models of wave propagation in damaged medium, it would be very convenient to have samples of material with controlled degradation. Application of a laser technology for material such as glass seems to be an interesting way to manufacture solid samples with well defined, small defects spread randomly, or in controlled way, in the bulk of material. Velocities of both longitudinal and shear ultrasonic waves in the glass are very similar to velocities in steel. SSLE technique enables to achieve various densities and orientation distributions of small cracks in the glass volume. Such defects can be a substitute of real cavities arising in the steel during creep. Experiments performed on several glass samples demonstrated that laser engraving produces material degradation measurable

with the ultrasonic technique and that obtained birefringence changes are similar to that obtained on the creep-tested steel samples. The elongated shape of laser-generated “pixels” forming small cracks is a reason of additional anisotropy observed in damaged glass samples. This anisotropy, caused by the nature of laser pixels, is seen when the shear wave propagates perpendicular to cracks plane.

It is worth mentioning that laser technology can be used to prepare glass samples with various other reflectors or artificial flaws. Experiments on few samples showed that randomly distributed “pixels” in the glass can also be used to imitate acoustic noise, obscuring signals during ultrasonic testing of coarse grain materials. Laser engraving can also be used to produce artificial flaws of any shape inside the glass block without any damage to its surfaces. Experiments showed that the surface built of many laser “pixels” is a good reflector for ultrasonic waves in low megahertz frequencies used in the ultrasonic nondestructive testing. Laser produced glass samples could also be used for UT training where artificial flaws can not only be ultrasonically detected, localized and measured but also directly visible for trainees.

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