

Investigation of Ultrasonic Properties of Hydrophilic Polymers for Dry-coupled Inspection

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Abstract. The use of dry-coupled ultrasonic techniques is of growing importance for inspection of components with non-uniform surfaces, advanced materials or coatings where the test piece material must not become wet or saturated with water. Rubber-like materials, often used for dry-coupled ultrasonic inspection, do not have the necessary ultrasound properties to make a good match between the transducer and the range of materials such as composites and ceramics since they exhibit high attenuation and can predominantly be used at low transmission frequencies. To overcome these problems, a number of different hydrophilic polymers with equilibrium water contents in the range 10 to 98 % by wet weight have been investigated at frequencies 1 MHz to 25 MHz. In the present study was investigated the change of ultrasonic velocity and attenuation in a temperature range from -30oC to 70oC which is important for infield inspection. The ultrasonic properties of hydrophilic polymers are studied and discussed with a view to the development of new dry-coupled probe. Demonstrating excellent acoustic properties, hydrophilic materials lend themselves very well to implementation in ultrasonic non-destructive testing. The results disclose their superiority over the most widely used ultrasonic couplants to transmit high frequencies when the testing procedure should meet specific requirements.

1. Introduction

Ultrasonic technique is one of the basic non-destructive methods for evaluation of materials and structures. A significant part of every ultrasonic inspection is the way in which the ultrasonic energy is transferred between the transducer and the tested object. Different types of commercial liquids and gels are used as a coupling medium. Sometimes the use of a liquid or gel couplant is undesirable because it may contaminate or penetrate into the material being tested leading to reduction of mechanical properties or corrosion. Instead of a liquid or gel couplant, rubber or synthetic rubber delay lines may be used for dry coupling. These materials in the form of pad, shoe or wheel are employed mainly for inspection in the kilohertz frequency range for highly attenuative materials such as plywood, ceramic and concrete [1-4]. In this low frequency range the coupling losses that occur are negligible. It is describe [5-11] the use of proprietary rubbers and different types of polymers with good acoustic properties in frequency range above 1MHz. For frequencies above 10MHz [12] it is also possible to use for dry-coupling thin polymer films stuck with vacuum [13].

Another way for conducting dry-coupled inspection at frequencies above 10 MHz is by using hydrophilic polymers [14]. They are truly dynamic systems that exhibit a singular balance of properties, which makes them appropriate for many applications. Hydrophilic

polymers are a unique group of plastic materials characterized by compatibility with water. Water acts as a plasticizer, and after swelling they transform from glass state to high-elastic rubber-like state. As with all polymers, their properties depend on their chemical composition and molecular structure. Poor mechanical properties of these materials can be enhanced by cross-linking to varying degrees [15]. Because of its high elasticity and flexibility this type of polymers conforms well to the rough and complex geometrical surfaces of the tested objects, by removing the air gaps and establishing reliable coupling without additional liquid. The swelling of high amount of water brings their acoustical properties near to those of water. This allows usage of frequencies above 10 MHz, as well as inspection automation without additional immersion liquid [14, 16 and 17].

This paper concentrates on the choice of an appropriate hydrophilic polymer for practical application in a newly designed device for dry-coupling contact ultrasonic inspection. This dry-coupled device will combine the advantages of inspection using focused ultrasonic transducer and dry-coupling. This device is easy to manipulate manually and gives flexibility of inspection in wide frequency and temperature range.

2. Experimental Samples and Set-up

Hydrophilic polymers characterized by different swelling factors were evaluated. Preliminary experiments show that polymers having water content above 85% can not be used as solid dry-couplants for practical purposes because of their low mechanical strength. Materials having water content under 30% are also not appropriate because their high sound attenuation and low flexibility. For this reason five materials presented in table 1 were investigated. They were designated as type-A to type-E and have water content in the range from 38% to 75%. The chosen polymers have similar mechanical strength which is needed for field inspection applications.

Table 1. Hydrophilic polymers

Type	Nature	Water Content at 20°C [%]
A	Poly hydroxy ethyl methacrylate	38
B	Copolymer of N-vinyl pyrrolidone and 2-hydroxy ethyl methacrylate	42
C	Poly hydroxy ethyl methacrylate	49
D	Terpolymer based on glycerol methacrylate	59
E	Copolymer of N-vinyl pyrrolidone and methyl methacrylate	75

In order to apply hydrophilic materials as dry-coupling medium it is necessary to study and compare their acoustical properties to those of other often used coupling materials. Samples were manufactured from rubber mixture based on polyisoprene rubber (type-F), especially synthesized proprietary elastomer material for acoustic couplant – Aqualene (type-G), and polymethylmethacrylate (PMMA) (type-H).

The hydrophilic polymers were machined in glass-state so as after swelling to become samples with different known thicknesses. From each type four different samples of polymers were produced having thickness 1mm, 3mm, 6mm, and 10mm in swelled state. For different samples the following symbols are used A, B...H. Symbols present the nature of the tested material and the numbers present their thickness. A3, for example, symbolizes 6 mm thick sample from Poly hydroxy ethyl methacrylate.

An immersion trough-transmission ultrasonic technique was used. This technique ensures steady and relatively slow change of the sample temperature and prevents the dehydration of the samples during investigation. The experimental setup used is shown on figure 1. The evaluations were carried out using two types of immersion liquids - distilled water and 96% ethyl alcohol. The temperature in the immersion tank was varied in the

range from 0°C to 70°C during experiments with distilled water and from -30°C to 30°C during ethyl alcohol experiments. Temperature was measured by K-Type thermocouple placed inside referent samples. Referent samples are from the same material and dimensions as the tested. Both the referent and the tested samples were positioned close to one another.

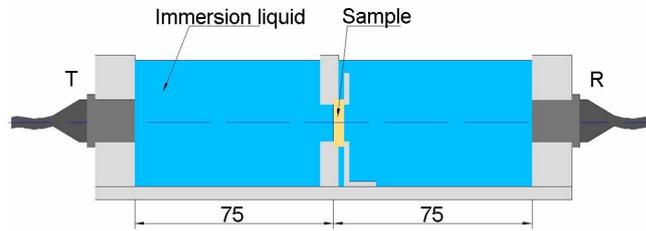


Fig. 1. Experimental setup

Experiments were done using spherically focused 10MHz transducers. For generation and recording of ultrasonic signals a computer based ultrasonic flaw detector PCIUT3100 (US Ultratek) with sampling frequency 100 MHz was used.

3. Results and Discussion

After testing the samples at different temperatures and immersion media the flowing results were obtained.

3.1 Acoustic Characteristics of Hydrophilic Polymers Swelled in Distilled Water at Room Temperature

Acoustic characteristics of tested materials are extremely significant when choosing an appropriate coupling medium and/or delay line. The longitudinal wave velocity is one of the important acoustic characteristics that affect the refraction angle when angle beam probe is used. It also affects the determination of the position of the registered flaw. For this reason the ultrasonic velocity is determined and compared with commonly used coupling mediums.

On figure 2 is presented the change of hydrophilic material's velocity depending on water content. When the water content increases the longitudinal ultrasonic velocity decreases and nears that of water – 1480 m/s. It is obvious that hydrophilic polymers have longitudinal ultrasonic velocity significantly lower than the velocity of PMMA (type-H) – 2750 m/s, polyizoprene rubber (type-F) – 1840m/s and is comparable to that of Aqualene (type-G) – 1580m/s. All types of hydrophilic polymers evaluated in this study demonstrated acoustic impedance's close to the impedance of water. As the acoustic mismatch on water/polymer surface is small a grater portion of energy is transmitted across this interface. Use of other materials leads to higher amount of reflected energy.

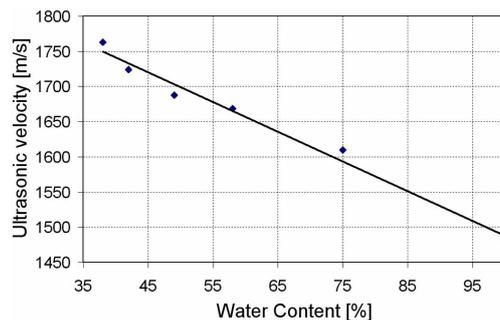


Fig.2. Longitudinal wave velocity vs. the water content in hydrophilic polymers.

It was expected that passing through the tested material some changes of signal spectrum could take place. The change in spectral characteristics of the signal transmitted through different hydrophilic polymers is presented on figure 4a, 4b, and 4c. The experiments are conducted using through-transmission technique and a 10 MHz transducer. Fourier transform on the signal was applied.

For the effective use of hydrophilic coupling mediums in industrial applications it is important to study the spectral characteristics of the signal transmitted through different hydrophilic polymers.

When the ultrasonic impulse passes through studied material its amplitude is reduced because of scattering and absorption of the ultrasonic energy and the high frequency components attenuate the most. The attenuation of the ultrasonic energy in hydrophilic polymers depends on the thickness of the sample as well as on the polymer's water content. For types (A-C) samples, where water content is low, a tendency of significant attenuation of high frequency components is observed and it depends on the sample thickness (fig. 4a). Polymers characterized by relatively high water content express a negligible change in the high frequency spectrum components when the thickness increases (fig.4c). The results for polymers with middle water content (type-D) is presented on figure 4b.

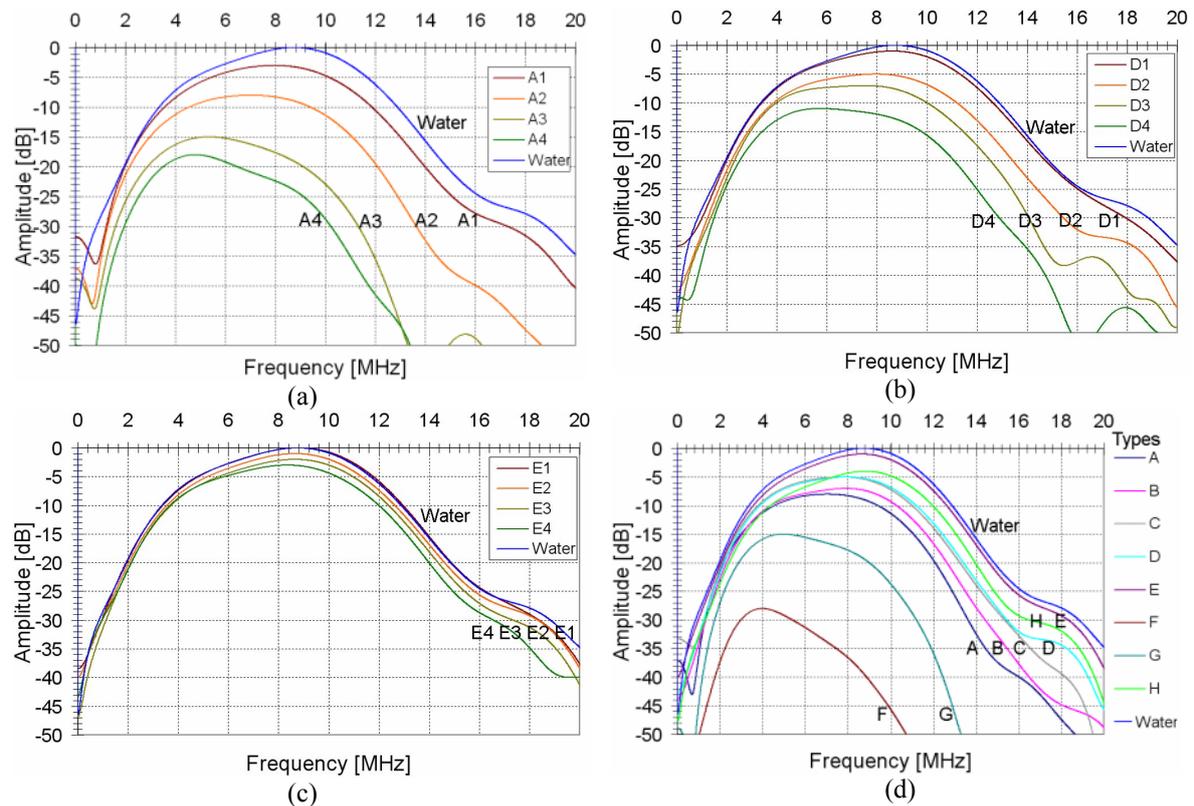


Fig.4. Frequency spectra of signals transmitted through the different types of investigated polymers

On figure 4d there are presented the signal spectra from all polymer types having equal thickness 3mm. The rubber type-F and Aqualene type-G are characterized by a significantly high attenuation compared to hydrophilic polymers. PMMA has an acoustical spectrum changes comparable to the water. Having more or less the same spectrum changes as PMMA the hydrophilic materials type-D and E could be preferred because of their higher surface flexibility.

The change of the central frequency of the spectrum and the bandwidth depending on the thickness were determined at -6 dB level (fig. 5 and 6). The samples having lower

water content (types A-C) have lower central- and lower peak-frequency as well as a steeper curve slope when thickness increases. The reason for this is the higher attenuation of the high frequency components in these samples. Greater changes in bandwidth and central frequency are observed for type-G and type-F materials. These polymers reduce significantly the frequency bandwidth of the signal which passes through them. They are not appropriate for frequencies above 6 MHz.

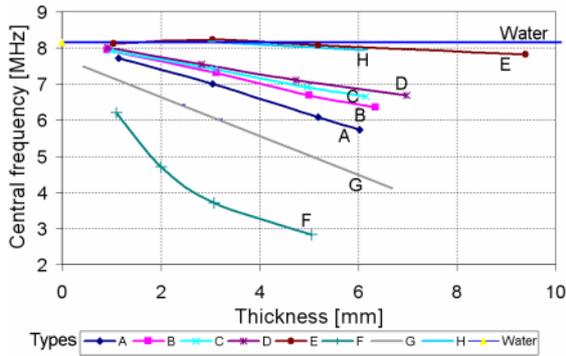


Fig.5. Change in the central frequency vs. thickness

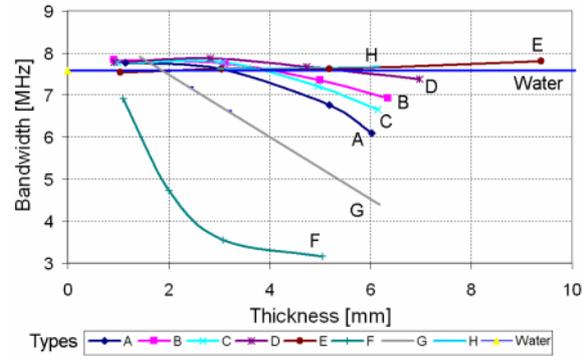
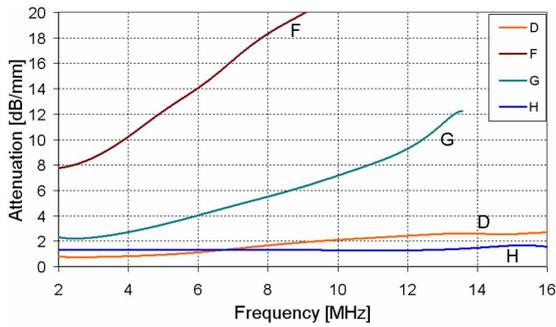
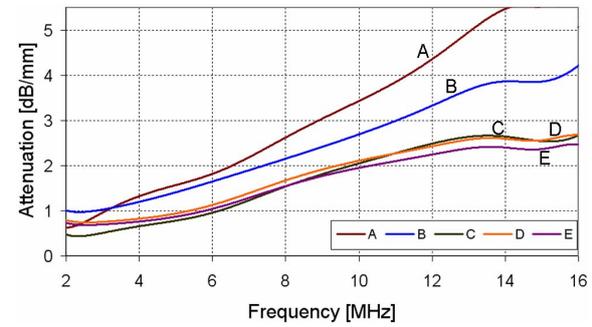


Fig.6. Change in the bandwidth vs. thickness

On figure 7a the attenuation was compared in rubber (type F), Aqualene (type G), PMMA (type H) and hydrophilic polymer type-D. The values of the rest of the hydrophilic materials are close to curve D and are presented on figure 7b. It was observed that the higher water content polymers (types D-E) assessed reduced attenuation compared to lower water content polymers. As the type-G and type-F materials attenuation is quite high it is better to use some of the hydrophilic polymers for an acoustic delay line.



(a)



(b)

Fig.7. Attenuation vs. frequency for different types of polymers

The results lead to the conclusion that hydrophilic polymers reveal significantly better acoustic properties at room temperature than the rest of the tested materials. The quantity of water in hydrophilic polymers affects the attenuation into these materials. The higher the water content the lower the effect on attenuation and spectrum changes.

3.2 Acoustic Characteristics of Hydrophilic Polymers Swelled in Distilled Water at Different Temperatures

The change in velocity of the longitudinal ultrasonic waves vs. change of temperature of the different types of hydrophilic polymers is presented on figure 8. The velocity of the longitudinal ultrasonic waves in types (A-B) decreases when the temperature increases. Conducted experiments show that such a tendency also exists in other polymer materials (elastomeric materials, PMMA, Aqualene). Since the samples of these two types contain less than 50 % water, their ultrasonic properties are closer to those of their base polymer. In

polymers with higher water content (types C-D) this tendency decreases, i.e. up to 35°C the velocity of the longitudinal ultrasonic waves slowly increases followed by decrease above this temperature. The change in the characteristics of the curve is most evident for polymers of type-E. The large quantity of water molecules in their structure brings their acoustic properties near to those of water, especially their longitudinal wave velocity.

As far as the change in the longitudinal ultrasonic velocity is concerned, hydrophilic polymers types C-D appear to be the most appropriate coupling medium in the temperature range from 0°C to 70°C. The reason for this is that the velocity of longitudinal ultrasonic waves changes negligible. In the temperature range from 15°C to 35°C the change is about 10 m/s.

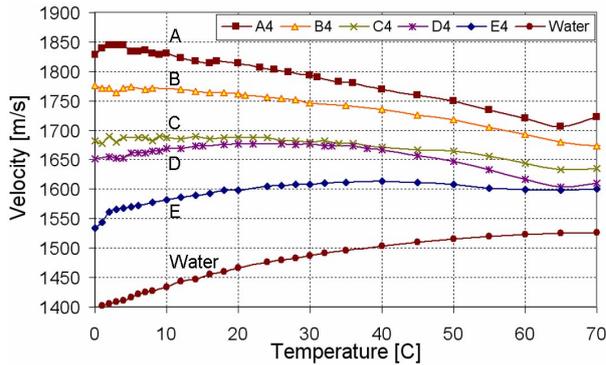


Fig.8. Ultrasonic velocity in different types hydrophilic polymers with temperature

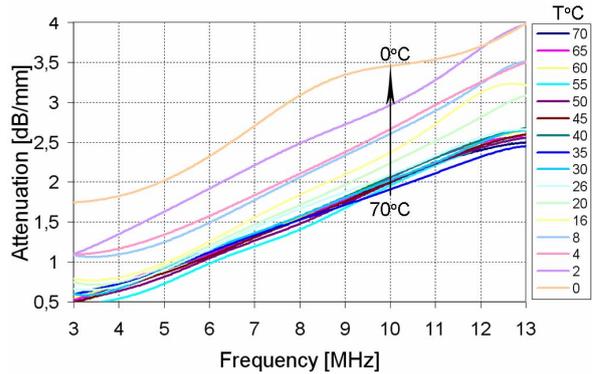


Fig.9. Ultrasonic attenuation in type-D with temperature

Ultrasonic attenuation in water and in different types of polymers also changes as a function of temperature. Experimental values of attenuation versus temperature for type-D polymers are presented on figure 9. Attenuation increases from 1,75 dB/mm@10MHz at 70°C to 3,5 dB/mm@10MHz at 0°C.

The change of the signal bandwidth and the skewness of the frequency spectrum depending on temperature are determined at -6dB level (figure 10 and 11). In hydrophilic polymers with lower water content the bandwidth of the signal spectrum increases with temperature while in types D-E it decreases and in the case of water it is constant. The lower water content polymers are more usable for temperatures above 35°C when types D and E polymers can be used in the temperature range from 5°C to 35°C.

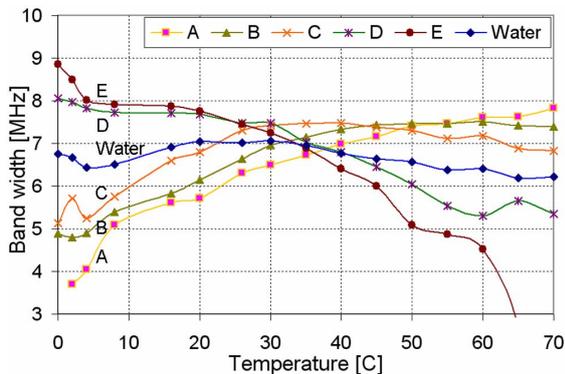


Fig.10. Change in spectrum bandwidth with change in temperature

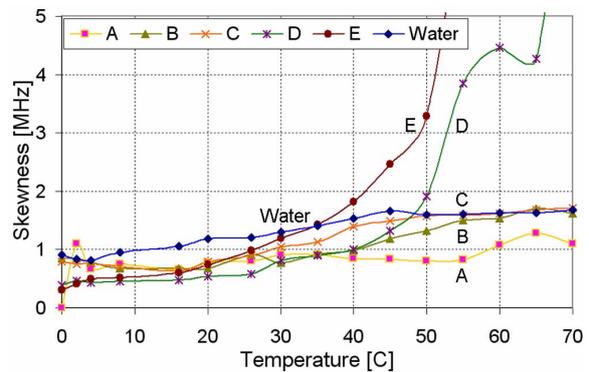


Fig.11. Skewness of spectrum with change in temperature

For most ultrasonic probes the spectrum curve shows a skewed rather than a Gaussian or normal shape. A measure of the amount of skew in spectrum offers another parameter that may be used to evaluate transducers performance. Skewness may be

represented by $f_{sk}=(f_p-f_a)/(f_b-f_p)$, where f_p is the peak frequency, f_a and f_b are the frequency locations corresponding to the -6dB level of the spectrum curve. The skewness of spectrum illustrated on figure 11 is determined for different types of hydrophilic polymers with the temperature change. High water content polymers types D-E exhibit a relatively high shift in the spectrum in temperatures above 35°C unlike types A-C where the change is slight.

The investigated temperature dependent acoustic characteristics are of significance for the choice of an appropriate hydrophilic material for infield applications where a wide range of working temperatures could be expected. From the conducted experiments can be concluded that type-D polymer is the most appropriate due to its good acoustic properties closest to those of type-E and water in the temperature range from 0°C to 35°C. At higher temperatures it is advisable to use type-C polymers. Another important criterion that makes the type D polymer superior to other tested materials is its structure allowing easy machining in different shapes. Besides, low dehydration velocity of these polymers presume longer usage period as a contact medium.

3.3 Acoustic Characteristics of Hydrophilic Polymers Swelled in Ethyl-Alcohol with Change in Temperature

Experiments with ethyl-alcohol as immersion liquid are conducted in order to achieve sub zero operation of the device [18]. Figure 12 presents the change in longitudinal wave velocity and figure 13 the signal bandwidth in temperature range from -30°C to +30°C. The change in velocity for all materials is about 15%. If these materials are to be used for oblique introducing of ultrasonic beam it will lead to change of incidence angle during testing.

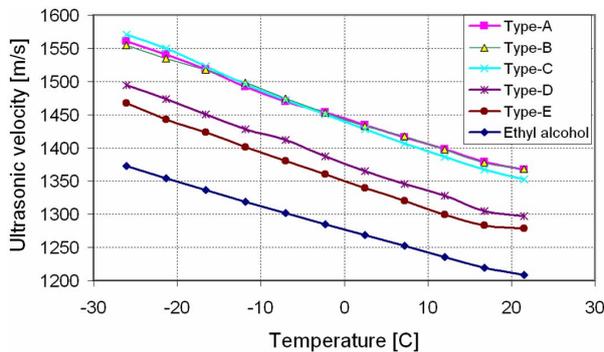


Fig.12 Ultrasonic velocity vs. temperature

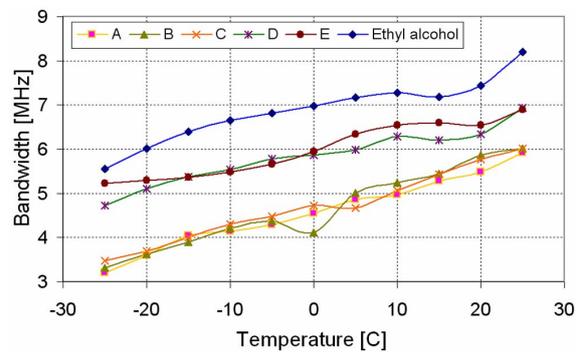


Fig.13 Bandwidth vs. temperature

Ultrasonic attenuation in ethyl-alcohol and in different types of polymers also changes as a function of temperature. Experimental values of attenuation vs. temperature for type-D polymers are presented on figure 14. Attenuation raises from 0,25 dB/mm@10MHz at 25°C to 1,75 dB/mm@10MHz at -25°C.

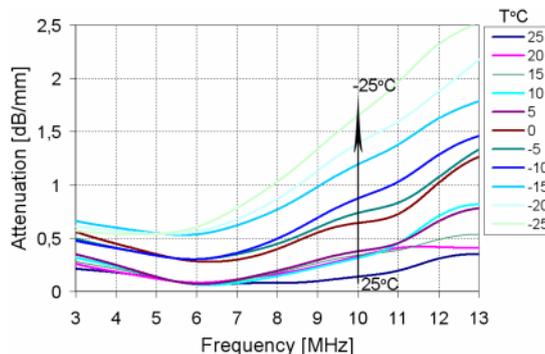


Fig.14. Attenuation vs. frequency for type-D polymers

Unlike other tested materials hydrophilic polymers can be used in a wide temperature range depending on the immersion liquid they are swelled in. At temperatures below -30°C rubbers lose their flexibility, unlike them hydrophilic polymers after absorption of some amount ethyl-alcohol remain flexible and can still established good acoustic contact.

On the bases of the conducted experiments the type-D hydrophilic polymer was chosen for the coupling material in an especially designed ultrasonic probe.

4. Application of Hydrophilic Polymers in Innovative Ultrasonic Probe

In the course of the experimental work on hydrophilic polymers an innovative ultrasonic contact probe has been designed and manufactured. It can be successfully applied in a range of industrial applications. The construction of the probe and the hydrophilic material protector is important for the establishment of a good acoustic coupling to the surface of the inspected product and for an effective transfer of ultrasonic energy.

The ultrasonic device shown on figure 15 comprises of a cylindrical body in the upper end of which a spherically focused ultrasonic transducer is mounted. The implemented ultrasonic transducer has a nominal frequency of 10 MHz. The focal length is 75 mm in water. The application of focused transducer helps to concentrate the beam energy as well as to overcome the dead zone and near field problems associated with the conventional probes. The transducer is mounted in a system allowing its axial movement in order to allow focusing at different depths in the tested part. This increases the possibilities for registration of flaws in the near surface region of the tested objects. In the lower end of the body a hydrophilic 7 mm delay line is mounted. The experiments show that this is the optimal thickness. The inner volume of the probe is filled with immersion liquid. In most cases distilled water is used as such liquid. The proposed design allows easy change of the ultrasonic transducer and the hydrophilic membrane, thus increasing the range of application of the proposed probe.

As the acoustic parameters of the hydrophilic layer are close to those of water it allows almost complete transmission of ultrasonic energy. The surface flexibility of the hydrophilic delay line allows the establishment of a good dry contact.

To demonstrate the advantages and excellent transmission of ultrasound of the proposed probe, experiments were carried out on parts, having different complex and surface roughness. It was established that the signal response from proposed device has 10dB smaller amplitude compared to the conventional immersion method. The proposed device was tested on different randomly rough surfaces up to $Rz\ 100\ \mu\text{m}$ and it was found that energy losses at high roughness are negligible.



Fig.15. Practical application of a proposed device and aluminium rough-plate

The ultrasonic signal recorded by the proposed device is compared on two surfaces – rough (Rz 90 μm) and nominally flat aluminium surface (Rz 4.28 μm). The back wall surface which is used to reflect the ultrasonic signal was milled to roughness Rz 4.28. Difference in back wall echo amplitude is about 6dB.

Additionally, the device was tested for thickness gauging applications. The achieved thickness resolution was about 0.8 mm which is comparable to commercially available gauge meters.

Conclusions

Considering the almost equal acoustic impedances of water and the proposed hydrophilic polymer, the energy reflected from the interface between them is negligible. The soft gel-like surface of the hydrophilic material gives the opportunity for establishing good and continuous acoustic contact to the inspected part without the need of additional coupling liquids. The proposed material emits a thin microfilm of immersion liquid over the inspected surface and conforms very well to rough and geometrically complex surfaces. Depending on the type of hydrophilic material used and its water content there exists an opportunity for transmission of ultrasonic impulses with frequency above 25 MHz. The shift and the bandwidth of the frequency spectrum emitted from the proposed device depend mainly on the acoustical characteristics of the hydrophilic polymer used and on the spectral composition of the signal emitted from the ultrasonic transducer.

The inspection of parts with randomly rough surfaces was demonstrated without the application of any additional coupling medium. The flexibility and elasticity of the used hydrophilic polymer allows the inspection of complex geometrical shapes and curved surfaces without the need to produce especially designed delay lines. Using different immersion liquids to swell the hydrophilic polymer the operating temperature range of the device can be extended from - 30° C up to 70° C.

References

- [1] Krautkraemer, J., H. Krautkraemer, „Ultrasonic Testing of Material”, Springer Verlag, 1990
- [2] Deutsch, V., M. Platte, M. Vogt, „Ultraschallprüfung – Grundlagen und industrielle Anwendungen”, Springer Verlag, 1997
- [3] Blitz, J., G. Simpson, „Ultrasonic Methods of Non-Destructive Testing”, Springer Verlag, 1996
- [4] Jones, M., G. Blessing and C. Robbins, „Dry-Coupled Ultrasonic Elasticity Measurements of Sintered Ceramics and Their Green States”, *Materials Evaluation*, Vol. 44, pp. 859-862, 1986
- [5] Ginzl, E., Ginzl, R., „Ultrasonic Properties of a New Low Attenuation Dry Couplant Elastomer”, *Ultrasonic Testing Online*, 1994
- [6] Robinson, A., B. Drinkwater, J. Allin, „Dry-coupled low-frequency ultrasonic wheel probes: application to adhesive bond inspection”, *NDT&E International* 36 (2003) 27–36
- [7] Ginzl, E., R. Ginzl, „The Ballprobe”, *NDTnet*, 1996, Vol.1 No.02
- [8] Drinkwater, B., R. Dwyer-Joyce, P. Cawley, „A study of the transmission of ultrasound across solid–rubber interfaces”, *The Journal of the Acoustical Society of America* Volume 101, Issue 2, pp. 970-981, 1997
- [9] Komsky, I, „Transducer Modules for Dry-Coupled Ultrasonic Inspection of Aircraft Structures”, *AIP Conference Proceedings – Feb. 26, 2004 - Volume 700, Issue 1*, pp. 713-720
- [10] Edwards, C., S. Dixon, S. Palmer, „Improvements to dry coupled ultrasound for wall thickness and weld inspection”, *AIP Conference Proceedings - May 23, 2000 - Volume 509, Issue 1*, pp. 1779-1786
- [11] Robinson, A., B. Drinkwater, „Extending the frequency range of the wheel probe—application to adhesive bond inspection”, *AIP Conference Proceedings, 2001, Vol. 557, 1*, pp. 883-890
- [12] Duwattez, J., F. Augereau, E. Caplain, J.M. Saurel, „Dry Coupling Ultrasonic High Frequency (10–100 MHz) Sensors for Detection of Surface and Tribological Properties at a Submicrometric Scale”, *Journal of Nondestructive Evaluation*, Vol. 22, No. 3, September 2003

- [13] Tohmyoh, H., M. Saka, „A Dry-Contact Method for Transmitting Higher Frequency Components of Ultrasound”, *International Journal of Applied Electromagnetics and Mechanics*, 18 (1-3), (2003), 31 - 39
- [14] Bourne, S., M. Newborough, D. Highgate, „Novel Solid Contact Ultrasonic Couplants Based on Hydrophilic Polymers”, 15th World Conference on NDT, 2000
- [15] Laporte, R., „Hydrophilic Polymer Coatings for Medical Devices”, CRC Press, 1997
- [16] Luprano, V., G. Montagna, B. Molinas, A. Maffezzoli, „Glass–rubber phase transformation detected in polymers by means of ultrasonic waves”, *Journal of Alloys and Compounds*, 310, 382–387 (2000)
- [17] Kutzarov, S., „Ultrasonic inspection of GRP Pipes using delay-line from hydrophilic material”, *Proceedings of the Scientific and Technical Union of Machine Building*, Year XII, Issue 1, 13-15 June, 2005
- [18] D'Arrigo, G., A. Paparelli, „Sound propagation in water–ethanol mixtures at low temperatures. I. Ultrasonic velocity”, *The Journal of Chemical Physics*, 1988, Vol. 88, Issue 1, pp. 405-415