Microelectronics Reliability 114 (2020) 113860

Contents lists available at ScienceDirect

Microelectronics Reliability

journal homepage: www.elsevier.com/locate/microrel

On the replacement of water as coupling medium in scanning acoustic microscopy analysis of sensitive electronics components

M. Hertl[a,*], F. Mialhe[b], I. Richard[a]

[a] Predictive Image, 193 rue Chassolière, 38340 Voreppe, France
[b] Centre Spatial de Toulouse, 18 av. E. Belin, 31055 Toulouse Cedex, France

ABSTRACT

The present paper studies the use of isopropyl alcohol and fluorocarbon liquids as coupling fluids for acoustic microscopy non-destructive testing of electronic flight components. For these components the standard coupling liquid water sometimes should be avoided in order to minimize sample contamination. The sound velocities of three different fluorocarbon liquids are measured, their ultrasound attenuation coefficients are estimated, and comparative failure analysis images on various electronics components are obtained by using water and these four alternative coupling liquids.

1. Introduction

Scanning acoustic microscopy (SAM) today is a standard means for non-destructive quality assessment and defect identification in electronics components and assemblies.

A particular case are the so-called flight model components in aeronautics and space industry. These components, integrated into satellites, space ships or aircraft, need to be extensively tested in order to achieve a high level of reliability. However, each SAM test involves immersion of the components into de-ionized water, which might be considered as a contaminant. In the ideal case, the used coupling liquid should be already part of the standard "life cycle" of the components, including manufacturing, test and screening. Natural candidates are isopropyl alcohol (used for cleaning) and fluorocarbon liquids such as Fluorinert™ FC-43, Galden® D02 and Galden® HT80 (used for seal testing following the MIL-STD-750 and the MIL-STD-883 standards), in spite of their known drawbacks such as fast evaporation and potential harmfulness to human health in case of isopropyl alcohol.

Few information is available in literature concerning the use of these fluids as acoustic coupling liquids. Even key parameters for the theoretical assessment of suitability such as sound velocity or attenuation constants are only partially known (cf. missing literature references in Tables 1 and 4).

Sound velocity values are well known for the standard coupling liquid water for temperatures from 0 °C to 100 °C [1]. For isopropyl alcohol and FC-43 some studies have been published, and values for the sound velocity at 20 °C have been reported [2,3] (see Table 1). To our knowledge, no literature data are available for D02 and HT80.

The sound attenuation by air-free distilled water at 20 °C is $\alpha/f^2 = 25 * 10^{-15}$ neper/m/Hz^2 [4]. Sound attenuation values in isopropyl alcohol have been reported at 20 °C: $\alpha/f^2 = 107 * 10^{-15}$ neper/m/Hz^2 (at 10 MHz) and $\alpha/f^2 = 86 * 10^{-15}$ neper/m/Hz^2 (at 1800 MHz) [5]. A value of $86 * 10^{-15}$ neper/m/Hz^2 (at 7 MHz, 21 °C) is reported in [6]. No quantitative acoustic attenuation data of FC-43, D02 and HT80 is available to our knowledge. Qualitatively, FC-43 is described as "highly attenuating, even at relatively low frequencies (10 MHz)" [7].

2. Experimental setup

Measurements are done with a commercial scanning acoustic microscope IS-350 manufactured by Insight kk (Japan). This instrument has a 4-axis (x,y,z, sample rotation) scanning mechanism. Samples are aligned parallel to the (x-y) working plane. The focussed ultrasound transducer T is mounted vertically to this plane along the z-axis, and can be moved along this axis for focussing the sound energy either onto the surface or into the volume of sample S (Fig. 1).

The sample and the transducer head are immersed into the coupling liquid CL. The liquid temperature is regulated and stabilized to values from 20 °C to 40 °C. The transducer is emitting a short sound pulse into the liquid. The sound pulse hits the sample, which might be either a flat glass plate or an electronics component, and is reflected back onto the transducer.

Ultrasound pulses with frequencies from 15 to 200 MHz are generated and their reflection received by a 500 MHz bandwidth pulser/receiver system (DPR500 from Imaginant Inc.). The ultrasound transducer (supplied by Insight kk) mainly used in this study has a central frequency of 15 MHz and a nominal focal length of 12.7 mm. For high resolution imaging, 35 MHz and 110 MHz transducers are used on

* Corresponding author.
E-mail address: m.hertl@predictiveimage.fr (M. Hertl).

https://doi.org/10.1016/j.microrel.2020.113860
Received 30 May 2020; Received in revised form 10 July 2020; Accepted 20 July 2020
Available online 31 October 2020
0026-2714/

Table 1
Sound velocities c at 20 °C measured for water, isopropanol, FC-43, D02 and HT80, and comparison with published data.

Liquid	Measured c (m/s)	Published c (m/s)
Deionized water	1482	1482 [1]
Isopropanol	1162	1156 or 1170 [2]
FC-43	673	658 [3]
D02	617	–
HT80	550	–

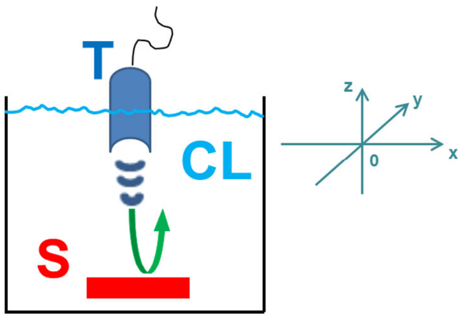

Fig. 1. Experimental setup. T: transducer. CL: temperature stabilized coupling liquid. S: sample.

specific samples.

The received sound pulse is converted into a high voltage signal and digitized by a PC (Fig. 2). The time of flight (TOF) of the pulse from the transducer to the sample and back depends on the transducer–sample distance and on the sound velocity in the coupling liquid. The peak amplitude depends on the transducer–sample distance, the sound attenuation by the coupling liquid, and the reflection and transmission coefficients of the involved liquid/sample interfaces.

3. Results and discussion

3.1. Sound velocity measurement in alternative coupling liquids

A thick glass plate is used as target for measuring the sound velocity

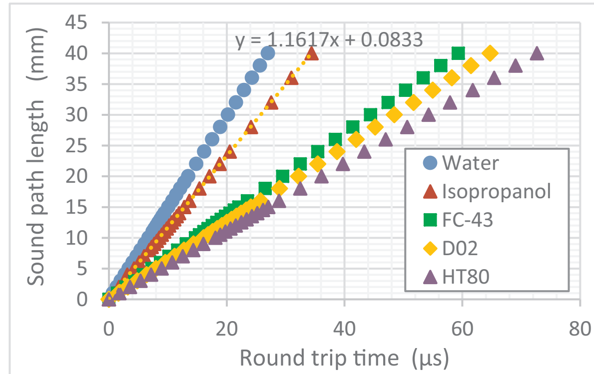

Fig. 3. Measurement of the sound path length vs. pulse round trip time in deionized water, isopropanol, FC-43, D02 and HT80, at 20 °C liquid temperature. The evaluation of the sound velocity by linear regression is shown for isopropanol.

of the tested coupling liquids. The transducer is moved up and down along the z-axis as shown in Fig. 1 by successive steps of 250 µm, 500 µm or 1000 µm by means of the z-axis stepper motor. For each z-position, the round-trip time of the pulse from the transducer to the glass plate and back to the transducer is measured. The negative peak TOF as shown in Fig. 2 is used for the time measurement.

Fig. 3 shows the result for the five coupling media deionized water, isopropanol, FC-43, D02 and HT80. For each set of data, the linear regression fit curve is calculated, as shown as an example for the isopropanol data set. The slope of the fit curve corresponds to the measured sound velocity in each of the coupling media. The standard deviation of the calculated slope is typically 0.2%. Table 1 shows the experimentally obtained sound velocities, together with previously published data.

The measured value for water perfectly matches the published one. The measured value for isopropanol is in between the two different published values. This result validates the accuracy of the experimental approach used in this paper.

The experimentally measured sound velocities for the three fluorocarbon liquids are significantly lower than those in water and isopropanol. The sound velocity value obtained in this study in FC-43 is

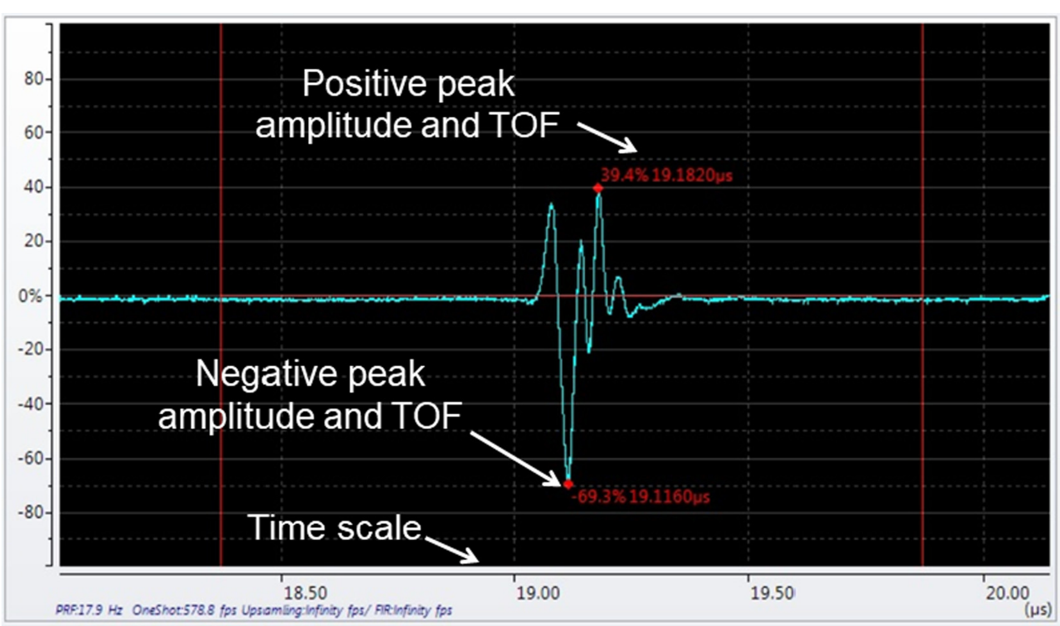

Fig. 2. Example of a received sound pulse. The round-trip time (time of flight – TOF) of the pulse from the transducer to the sample and back is displayed on the abscissa. The ordinate shows the peak amplitude as percentage of the full scale.

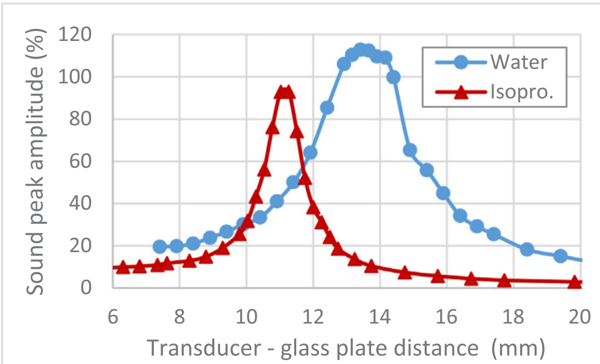

Fig. 4. Reflected ultrasound amplitude vs. transducer-to-glass plate distance with water and isopropanol as coupling liquids. The nominal focal length of the transducer in water is 12.7 mm.

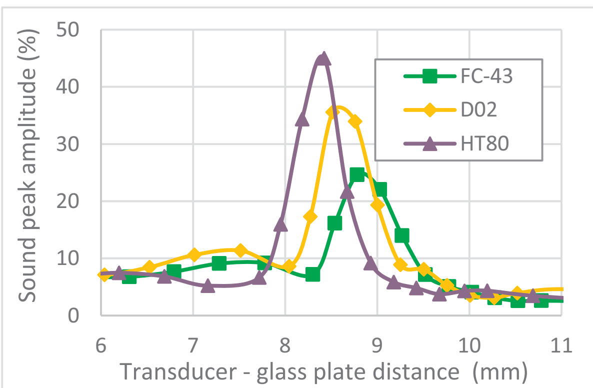

Fig. 5. Reflected ultrasound amplitude vs. transducer-to-glass plate distance with FC-43, D02 and HT80 as coupling liquids. The nominal focal length of the transducer in water is 12.7 mm.

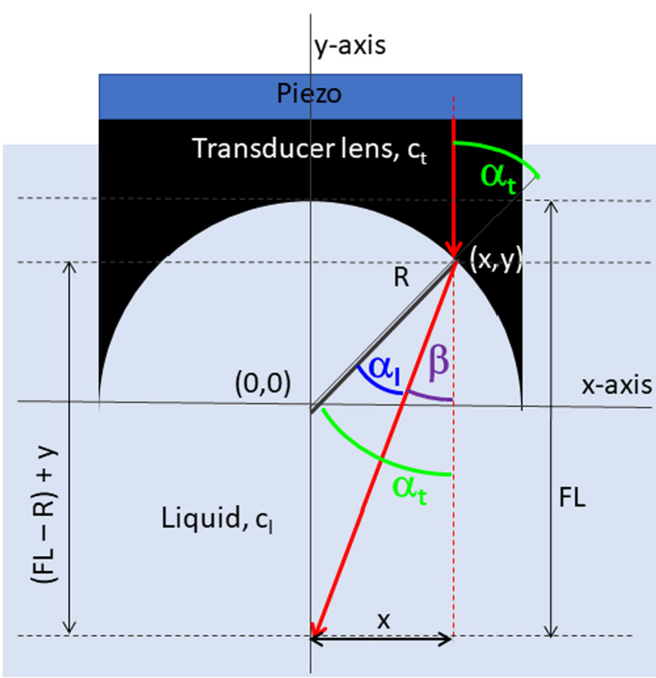

Fig. 6. Schematic representation of a plane sound wave, travelling through the transducer lens and being focused by the curved lens-liquid interface. c_t, c_l: sound velocities in the transducer lens and the coupling liquid, respectively.

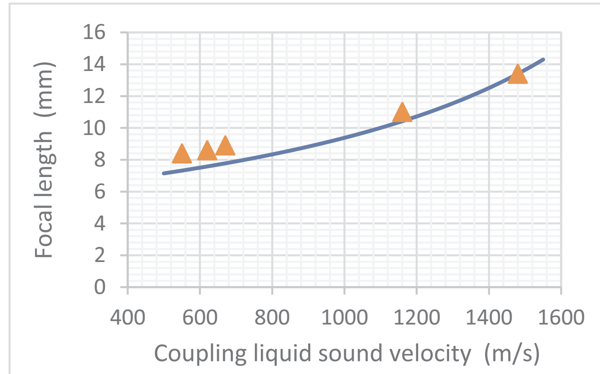

Fig. 7. Calculated variation of the focal length of the used 15 MHz transducer (*FL* 13.4 mm in water) with the coupling liquid's sound velocity (line). An estimated sound velocity of 2600 m/s is used for the transducer's epoxy lens. Markers: experimentally determined focal lengths with the five coupling liquids used in this study.

Table 2
Focal length of the used FL 13.4 mm transducer in the five coupling liquids as determined experimentally (Figs. 4, 5) and estimated theoretically (Fig. 7).

Liquid	*c* (m/s)	Experimental *FL* (mm)	Calculated *FL* (mm)
Water	1482	13.4	(13.4 set)
Isopropanol	1162	11.0	10.4
FC-43	673	8.9	7.8
D02	617	8.6	7.6
HT80	550	8.4	7.3

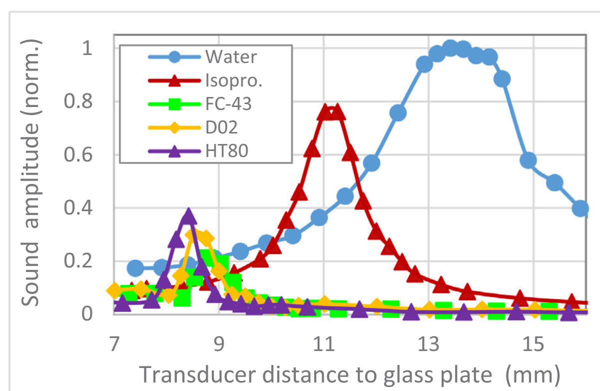

Fig. 8. Reflected ultrasound amplitude vs. transducer-to-glass plate distance for the five coupling liquids of this study. For each liquid, the measured sound amplitude is divided by the reflection coefficient of the sound on the respective liquid/glass interface.

slightly higher than the value reported in [3]. No comparative data are available for D02 and HT80.

3.2. Focussing in alternative coupling liquids

Knowing the influence of the coupling liquid on focussing and attenuation of the ultrasound energy emitted from the transducer towards the sample is of primary importance for the preparation of the SAM test setup for a given sample.

The numerical values for the focal length of ultrasonic transducers with an integrated acoustic lens for focussing are only valid if the transducers are used in water. It depends on the curvature of the transducer lens and on the sound velocities in the lens material and the coupling medium. If a coupling medium other than water is used, the indicated focal length is no longer valid.

Table 3
Acoustic impedance of the coupling liquids and reflection coefficients of sound waves on the respective liquid/glass interfaces, at 20 °C. The experimental sound velocities of Table 1 are used.

Liquid	ρ (kg/m^3)	$Z = c * \rho$ 10^6 Nm/s	R_l (liquid/glass)
Water	998.2 [9]	1.479	81.5%
Isopropanol	785 [10]	0.912	88.2%
FC-43	1869 [11]	1.258	84.0%
D02	1782 [12]	1.099	85.9%
HT80	1690 [13]	0.930	87.9%

Table 4
Estimated attenuation coefficients at 20 °C for isopropanol, FC-43, D02 and HT80, and comparison with published data.

Liquid	Measured α/f^2 (10^{-15} neper/m/Hz^2)	Published α/f^2 (10^{-15} neper/m/Hz^2)
Water	25 (reference)	25 [4]
Isopropanol	82	107 [5], 86 [6]
FC-43	427	–
D02	346	–
HT80	303	–

For studying the focussing and attenuation of the ultrasound beam the transducer was moved along the z-axis, with a glass plate as target. At each position the acoustic amplitude is noted. Figs. 4 and 5 show the reflected ultrasound amplitudes vs. the transducer-glass plate distance for the five coupling liquids used in this study. In both Figures the same y-axis scale is used, making the obtained amplitudes immediately comparable.

In case of water maximum signal amplitude is reached for a transducer distance of 13.4 mm from the glass plate, which is reasonably close to the transducer's nominal focal length. For isopropanol, FC-43, D02 and HT80 the peak intensities are reached for successively lower transducer-to-glass distances, as a consequence of the lower sound velocities in these coupling liquids.

As a simplified model we consider the sound wave from the piezo crystal through the transducer lens into the coupling liquid as an initially plane wave with sound velocity c_t in the transducer lens and c_l in the coupling liquid. In a coordinate system with its origin on the transducer axis as shown in Fig. 6, the part of the sound wave hitting the transducer-liquid interface at (x,y) will be refracted following Snell's law, which determines the angle β by

$$\sin\beta = \frac{x}{R}\frac{c_l}{c_t}\left\{\sqrt{\left(\frac{c_t}{c_l}\right)^2 - \left(\frac{x}{R}\right)^2} - \sqrt{1 - \left(\frac{x}{R}\right)^2}\right\}.$$

Knowing β allows to calculate the focal length FL as

$$FL = \frac{x}{\tan\beta} + R - \sqrt{R^2 - x^2}$$

In case of a small aperture lens (x ≪ R) the approximation

$$\lim_{x\to 0}\sin\beta = \lim_{x\to 0}\tan\beta = \beta$$

allows to find

$$FL = R\,\frac{c_t}{c_t - c_l} \tag{1}$$

With FL_w and c_w being the transducer's focal length and the sound velocity in water, the focal length of the same transducer in any other liquid can be estimated as

$$FL = FL_w\,\frac{c_t - c_w}{c_t - c_l} \tag{2}$$

Fig. 7 shows the focal length calculated by using Eq. (2) for the 15 MHz transducer used in this study to investigate coupling liquids with sound velocities ranging from 500 m/s to 1500 m/s, together with the experimentally obtained values. Table 2 shows a numerical comparison of the theoretical and experimental values for the five tested coupling liquids.

The experimental and calculated results show the same trend: the transducer's focal length decreases with decreasing sound velocity in the coupling medium. However, the calculation slightly overestimates the decrease of the focal length with the liquid's sound velocity. This is probably due to the consideration of simple diffraction following Snell's law on the transducer/liquid interface. In reality, even transducers without a focussing lens generate a somewhat focussed sound beam, with the focus point at the transition from near to far field. Therefore, the radius of the epoxy lens of our transducer should be larger than calculated by Eq. (1), and the total focus length of the transducer should include a term that takes into account the "natural" focussing of the sound by the transducer aperture, without presence of any lens at the transducer tip.

3.3. Attenuation in alternative coupling liquids

For visualization of the acoustic attenuation Fig. 8 shows again the reflected sound amplitude vs. transducer-to-glass plate distance. Differently to Figs. 4 and 5, the graphs in Fig. 8 are corrected for the

Fig. 9. The samples used in this study are reference samples for long-term reliability analysis at French national space research center CNES. From left to right: FlipChip, PQFN208, TO.

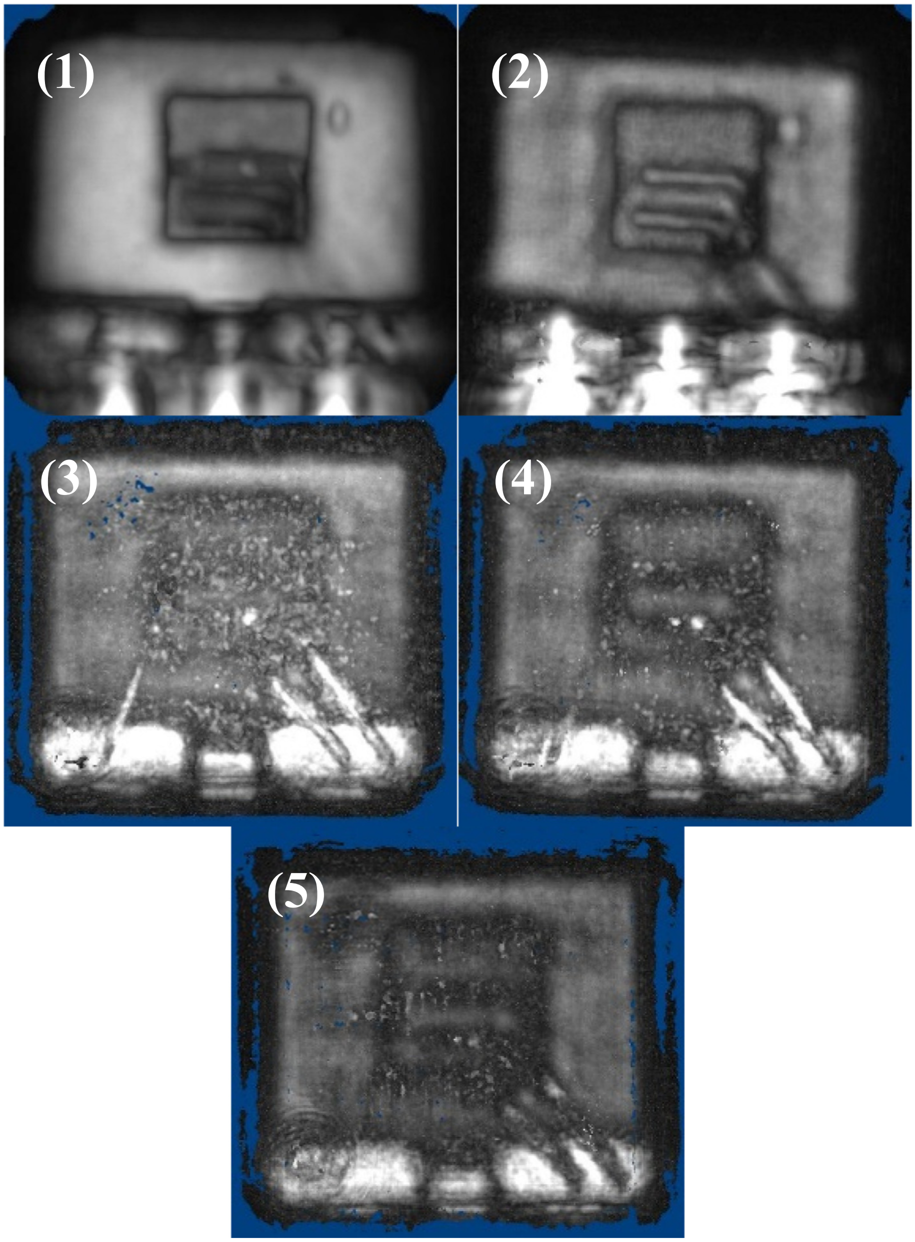

Fig. 10. Top side analysis of the TO component by a 15 MHz transducer, in water (1), isopropanol (2), FC-43 (3), D02 (4) and HT80 (5) at 20 °C.

reflection coefficient of the sound on the glass plate, calculated by

$$R_l = \frac{Z_g - Z_l}{Z_g + Z_l}$$

where $Z_g = 14.5 * 10^6$ Nm/s is the acoustic impedance of crown glass [8] and Z_l the acoustic impedance of the coupling liquid. Reflection coefficients used in this study are summarized in Table 3.

Due to the higher attenuation of isopropanol compared to water, the maximum measured amplitude for this liquid is lower. As expected, the sound attenuation in the fluorocarbon liquids is even higher as in isopropanol, resulting in significantly lower received sound amplitudes.

Quantitatively, the sound amplitude (voltage) received by the transducer can be expressed as

$$I_l = I_0 \, R_l * e^{-\left(\frac{\alpha_l}{f^2} * f^2 \, d_l\right)} \tag{3}$$

where I_l is the received sound amplitude in the coupling liquid, I_0 the initially emitted sound amplitude, α_l the attenuation coefficient of the coupling liquid, f the transducer frequency, and d_l the sound path (transducer to glass plate and back) in the coupling liquid. Note that the attenuation coefficient divided by the square of the frequency is used in the equation, as this is the usually referenced attenuation value [4–6].

Using the well-known attenuation coefficient of water of $\alpha_w/f^2 = 25 * 10^{-15}$ neper/m/Hz^2 [4] allows to estimate the attenuation coefficients α_l of the other coupling liquids by using Eq. (3) together with experimentally obtained sound amplitude ratios I_l/I_w with the transducer focused on the glass plate:

$$\frac{\alpha_l}{f^2} = \frac{\frac{\alpha_w}{f^2} f^2 \, d_w - \ln\left(\frac{I_l/R_l}{I_w/R_w}\right)}{d_l \, f^2} \tag{4}$$

The graphs in Fig. 8 are normalized with respect to the reflection coefficient on the glass plate for each liquid. Thus, for each liquid, the intensity ratio I_l/R_l needed in Eq. (4) corresponds to the peak value of the curve for this liquid, and the value d_l corresponds to the double of the transducer's focal length in this liquid.

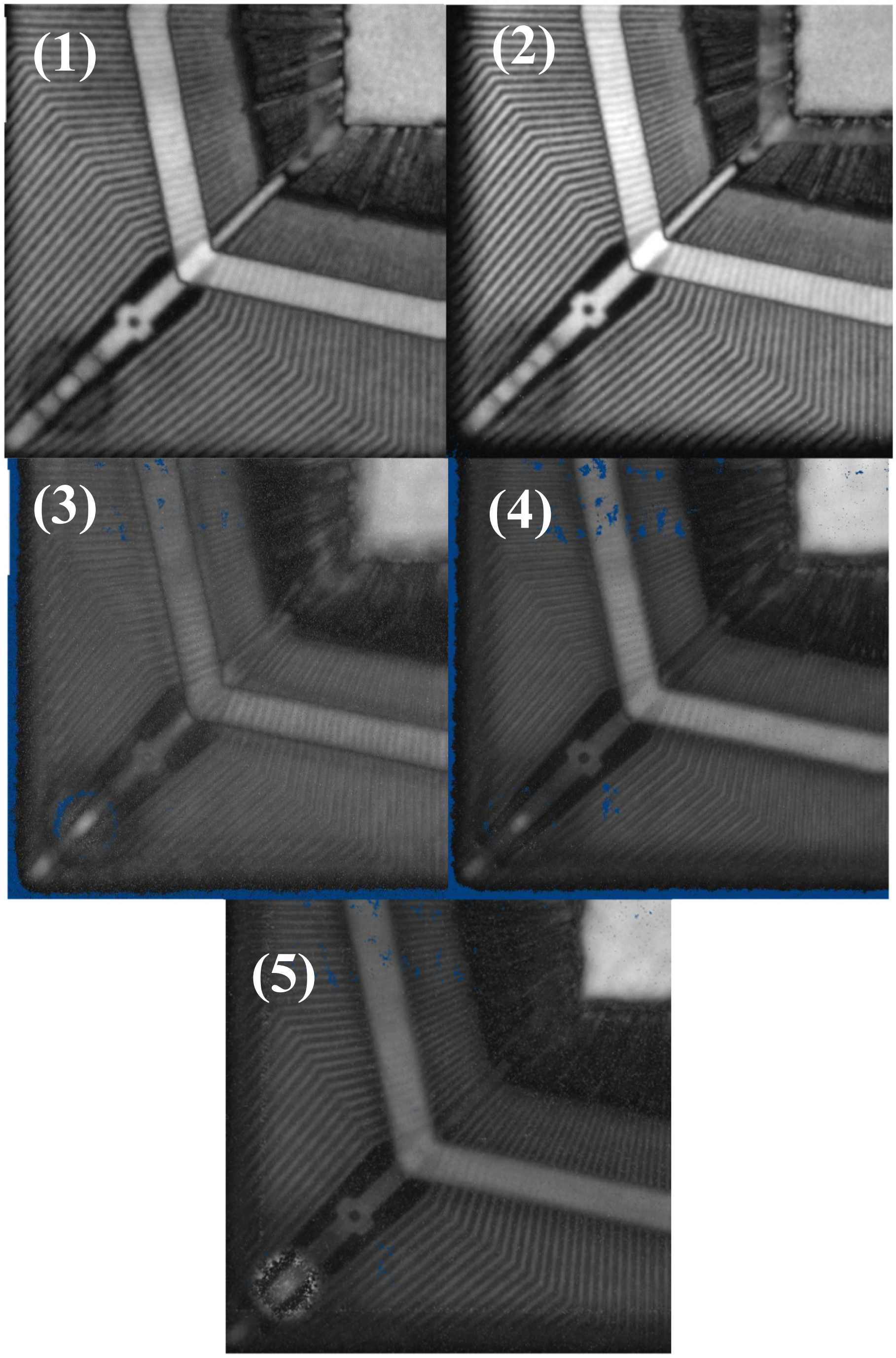

Fig. 11. PQFN analysis in water (1), isopropanol (2), FC-43 (3), D02 (4) and HT80 (5) at 20 °C, using a 35 MHz transducer.

Table 5
Estimated beam diameter in the focal spot of a transducer with f = 15 MHz, FL (water) = 13.4 mm, D = 6.3 mm in the five coupling liquids.

Liquid	BD_{-6dB} (μm)
Water	294
Isopropanol	189
FC-43	89
D02	79
HT80	68

Table 4 shows the estimated attenuation coefficients at 20 °C and 15 MHz transducer frequency. These values should be considered as estimations rather than exact values, due to some important approximations done in the calculation. In particular, it is considered that the same fraction of reflected sound energy is collected by the transducer for all five coupling liquids, which is questionable due to the variation of the transducer–glass plate distance. Also, equal coupling of sound energy between the transducer tip and the liquid is supposed for all liquids.

However, the estimated attenuation coefficient for isopropanol agrees well with previously published data, which seems to confirm the overall correctness of the applied approach. The attenuation coefficients deduced similarly for fluorocarbon liquids show the lowest value for the Galden HT80 liquid, and the highest sound attenuation for

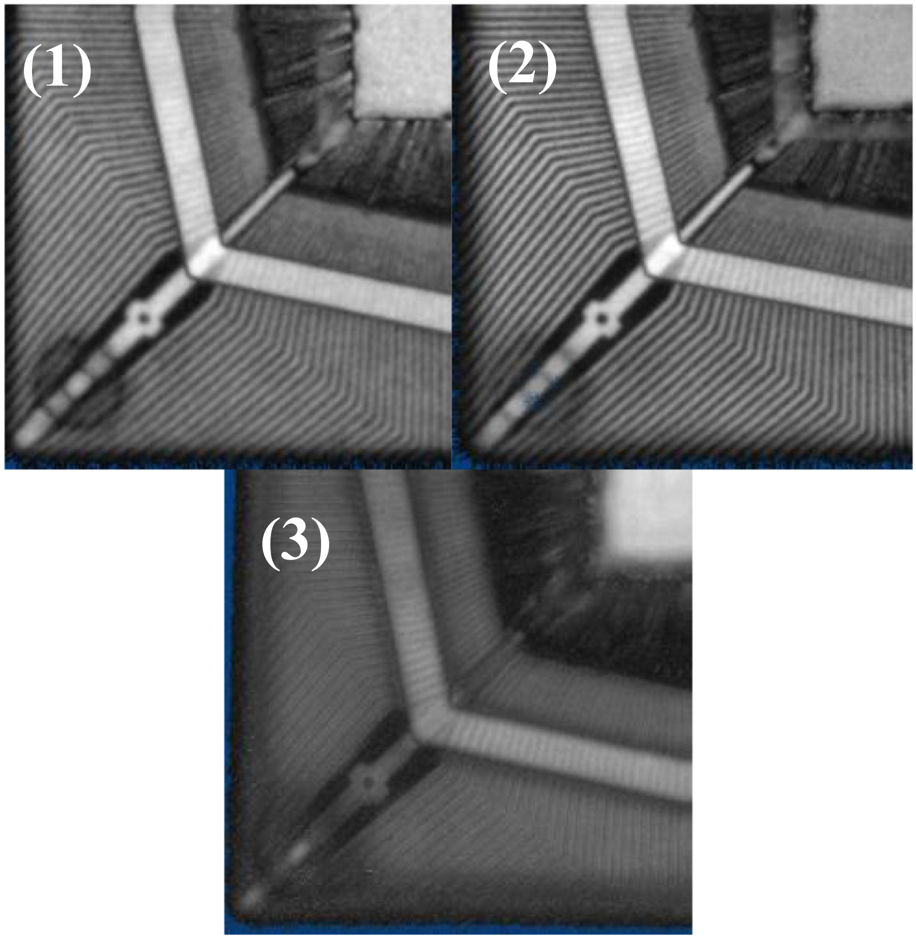

Fig. 12. PQFN208 analysis in water (1), isopropanol (2) and FC-43 (3) at 40 °C, using a 35 MHz transducer.

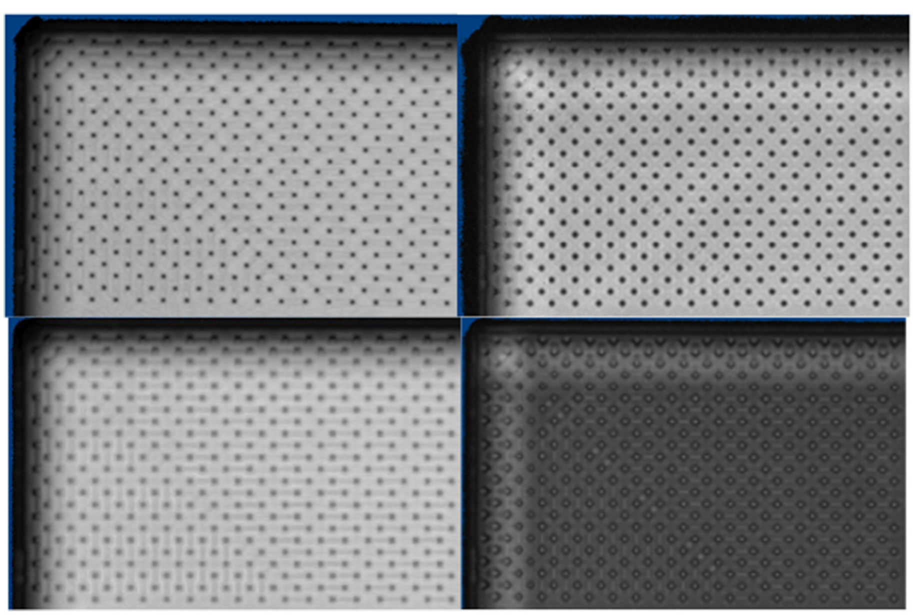

Fig. 13. FlipChip analysis in water (left) and isopropanol (right) at 20 °C (top) and 40 °C (bottom) with a 110 MHz transducer.

Fluorinert FC-43.

3.4. Flight components analysis in alternative coupling liquids

The influence of the coupling medium and its temperature on SAM images of electronic components has been studied for three different component types (Fig. 9) at liquid temperatures ranging from 20 °C to 40 °C. Various ultrasonic transducers with frequencies from 15 to 110 MHz have been used.

Figs. 10 and 11 show respectively SAM images of the TO and the

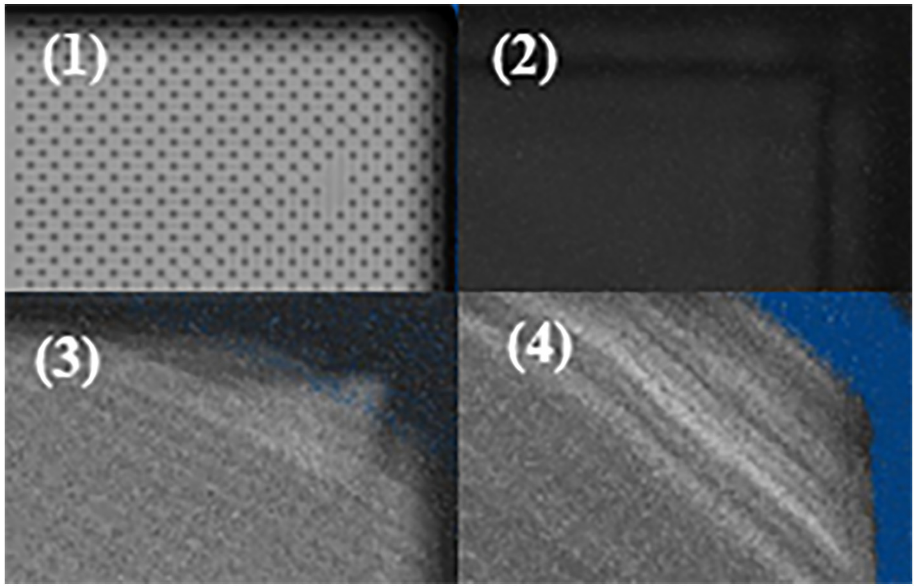

Fig. 14. FlipChip analysis in water (1), FC-43 (2), D02 (3) and HT80 (4) at 20 °C, using a 110 MHz transducer.

PQFN sample with the five different coupling liquids, at 20 °C. For both samples, similar observations can be made:

Both water and isopropanol show comparable image quality, with the image in isopropanol being slightly sharper and better defined in both cases. This behaviour can be explained by the lower sound velocity in isopropanol, and also the shortening of the focal length of the 15 MHz transducer when used in isopropanol instead of water (the 35 MHz transducer is focussed by its curved element, and therefore does not show a variation of focal length when used in different liquids). The image resolution is directly related to the beam diameter $BD_{-6\mathrm{dB}}$ in the focal spot of the acoustic beam, which depends on the frequency f, the piezo diameter D, the transducer's focal length FL and the sound velocity c in the coupling liquid [14]:

$$BD_{-6\,\mathrm{dB}} = 1.4 * \frac{c * FL}{f * D}. \tag{5}$$

As an example, Table 5 shows calculated focal spot diameters for the used 15 MHz transducer based on the experimentally determined sound velocities and focal length parameters.

However, it has to be considered that in strongly absorbing liquids the attenuation, which is proportional to the square of the frequency, acts as a kind of frequency shifter. Therefore, for isopropanol, but in particular for the fluorocarbon liquids, the acoustic frequency reaching the sample is substantially lower than the frequency emitted by the transducer. Using the initial transducer frequency in Eq. (5) will overestimate the lateral resolution on the sample in these liquids. For fluorocarbon liquids, the frequency shift by strong attenuation annihilates the benefits of the shorter focal length.

Fig. 12 shows the same images as Fig. 11, but acquired with a liquid temperature of 40 °C. For this 35 MHz transducer, the image quality stays about the same for the increased temperature, in particular in water and isopropanol.

At higher frequencies, the temperature influence on the resulting image becomes more important, as can be seen in Fig. 13 for the example of a FlipChip analysis at 110 MHz. At this frequency, when increasing the temperature, images acquired in water become more intense, but somewhat blurred. On the other hand, images acquired in isopropanol become less intense, but better defined, with more details visible.

The intensity increase with temperature in water is an expected result, as the sound attenuation of water decreases with increasing temperature [4]. However, the same observation is to be expected for isopropanol, which also has a decreasing sound attenuation with increasing temperature [5].

For increasing temperature, the sound velocity increases in water, but decreases in isopropanol. Following Eq. (5), we would therefore expect, for increasing temperature, a decrease of resolution in water and an increase of resolution in isopropanol, which is obviously the case.

At high frequencies such as 110 MHz, the fluorocarbon liquids become unusable for SAM applications, even at low temperatures, as illustrated on Fig. 14, due to their high attenuation coefficients.

CRediT authorship contribution statement

M. Hertl:Conceptualization, Methodology, Validation, Formal analysis, Investigation, Data curation, Writing - original draft, Writing - review & editing, Visualization.**F. Mialhe**:Conceptualization, Methodology, Validation, Resources, Writing - review & editing, Supervision, Project administration, Funding acquisition.**I. Richard**:Conceptualization, Methodology, Resources, Writing - review & editing, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References

[1] Greenspan, et al., J. Res. Natl. Bur. Std. 59 (4) (Oct. 1957).
[2] The Dortmund Data Bank (DDB), Online http://www.ddbst.com/en/EED/PCP/SOS_C95.php.
[3] Sturtevant, et al., J. Acoust. Soc. Am 138 (1) (July 2015).
[4] G.W.C. Kaye, T.H. Laby, Tables of Physical and Chemical Constants, Longman, New York, 1995.
[5] Bulavin, et al., J. Mol. Liq. 235 (June 2017) 24–30.
[6] Wallace, et al., Ultrasound Med. Biol. 33 (4) (2007 Apr) 620–629.
[7] Blake Sturtevant, Los Alamos National Laboratory, MPA-11 MS D429, Los Alamos, NM87545, private communication (2018).
[8] Accoustic Properties for Ceramics, NDT resource center, Retrieved May 25, 2020 from https://www.nde-ed.org/General/Resources/MaterialProperties/UT/ut_matlprop_ceramics.htm.
[9] Internetchemie.info, Die naturwissenschaftliche Wissensdatenbank, Retrieved May 25, 2020 from http://www.internetchemie.info/chemie-lexikon/daten/w/wasser-

dichtetabelle.php.
[10] PubChem, Isopropyl alcohol, Retrieved May 25, 2020 from https://pubchem.ncbi.nlm.nih.gov/compound/Isopropyl-alcohol#section=Density.
[11] Fluorinert™ electronic liquid FC-43. Product information. 3M, Retrieved May 25, 2020 from https://multimedia.3m.com/mws/media/64889O/fluorinert-electronic-liquid-fc-43.pdf.
[12] Hirtz F, Kurt J. Lesker Co Ltd., 15/16 Burgess Road, Hastings, East Sussex, TN35 4NR : (Private communication).
[13] Galden® PFPE, Product information. Solvay, Retrieved May 25, 2020 from https://www.solvay.com/en/brands/galden-pfpe.
[14] Insight kk: Private communication (2016).