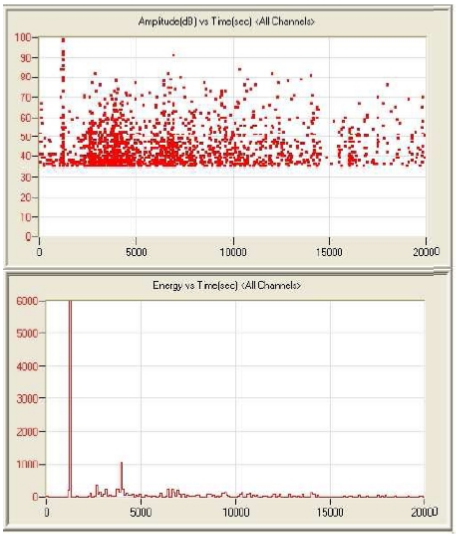
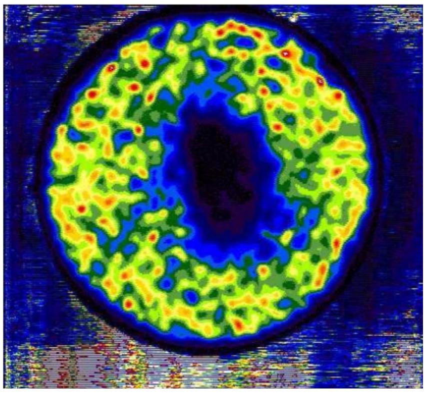
Nationaal Lucht- en Ruimtevaartlaboratorium
National Aerospace Laboratory NLR

Executive summary

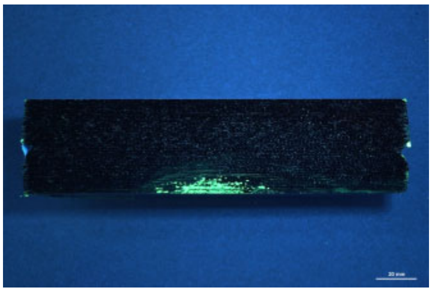
Acoustic emission and ultrasonic inspection of cure related defects in thick RTM products



AE activity (amplitude and energy versus time) of RTM product 2149 during cooling down to room temperature



Ultrasonic C-scan of RTM disc 2149



Improper impregnation in the central product region opposite the vent hole location

Problem area
The resin transfer moulding (RTM) technique is increasingly applied for the fabrication of thick complex shaped and highly loaded components in aerospace composite structures (wall thickness exceeding 50 mm). Higher volumes of RTM material, however, may decrease the product performance due to cure related material degradation like the formation of shrinkage defects and porosity. For the fabrication of good quality thick RTM products there is hence a need for non-destructive inspection (NDI) techniques for quality control and to optimise the process window for RTM.

Description of work
RTM discs and plates of different thickness were manufactured. A range of RTM processing variables were herewith varied. The acoustic emission (AE) technique was applied for defect monitoring during the cooling down phase of the RTM product to room

Report no.
NLR-TP-2006-323

Author(s)
J.H. Heida
W.G.J. 't Hart

Classification report
Unclassified

Date
June 2006

Knowledge area(s)
Levensduurbewaking en onderhoud van vliegtuigen

Descriptor(s)
acoustic emission
nondestructive
ultrasonic
RTM

This report is based on a presentation to be held on the 9th European Conference on Non-Destructive Testing, Berlin, 25-29 September 2006.

temperature, and the ultrasonic C-scan technique was used to assess the final material quality of the RTM product.

Results and conclusions

The results of the investigation show a relation between AE activity, UT attenuation level and the presence of cure related defects in RTM products. A higher level of AE activity corresponds in general with a higher ultrasonic attenuation level and a lower quality of the RTM product. The following trends were observed when studying the influence of RTM material and process parameters on the AE activity and final material quality:

- A brittle resin system generally results in higher AE activity in terms of number of AE hits.
- An RTM mould of special design instead of a fixed wall results in a significant reduction of the AE activity and in a higher material quality.
- A lower heating rate results in a reduction of the AE activity and in a somewhat higher material quality.

Applicability

The present investigation shows that the AE and UT technique can be used to monitor the quality of RTM products during the fabrication and after production. During the investigation too many RTM processing parameters were varied. Therefore, the exact role of different cure related defect types on the AE activity remains to be established in a further investigation.

Nationaal Lucht- en Ruimtevaartlaboratorium, National Aerospace Laboratory NLR

Anthony Fokkerweg 2, 1059 CM Amsterdam,
P.O. Box 90502, 1006 BM Amsterdam, The Netherlands
Telephone +31 20 511 31 13, Fax +31 20 511 32 10, Web site: www.nlr.nl

Nationaal Lucht- en Ruimtevaartlaboratorium

National Aerospace Laboratory NLR

NLR-TP-2006-323

Acoustic emission and ultrasonic inspection of cure related defects in thick RTM products

J.H. Heida and W.G.J. 't Hart

This report is based on a presentation to be held/held on the 9th European Conference on Non-Destructive Testing, Berlin, 25-29 September 2006.

Customer	National Aerospace Laboratory NLR
Contract number	---
Owner	National Aerospace Laboratory NLR
Division	Aerospace Vehicles
Distribution	Unlimited
Classification of title	Unclassified

Approved by:

Author	Reviewer	Managing department
23/06/06	W 't Hart 23/06/06	16/8

Summary

This paper describes an investigation into the applicability of the acoustic emission (AE) and ultrasonic inspection (UT) technique for the monitoring of cure related defects in thick resin transfer moulded (RTM) products. The AE technique was applied for defect monitoring during the cooling down phase to room temperature while the UT technique was used to assess the final material quality of the product. The results of the investigation show a relation between AE activity, UT gain level and the presence of cure related defects such as shrinkage cracks and porosity. A higher level of AE activity corresponds in general with a higher UT attenuation level and a lower quality of the RTM product. The results of the investigation were used to detail the influence of RTM material and process parameters such as resin type (tough, brittle) and heating rate during RTM processing. The results were further used to optimise the design of the RTM product mould.

Contents

2 Tables

15 Figures

(23 pages in total)

Abbreviations

AE	Acoustic Emission
G_{IC}	Strain energy release rate [J/m^2]
HTS	High Tensile Strength
K_{IC}	Fracture toughness [MPa.m^{½}]
η	Viscosity at T_i [mPa.s]
NCF	Non-Crimp Fabric
PAC	Physical Acoustics Corporation
RT	Room Temperature
RTM	Resin Transfer Moulding
T_c	Curing Temperature [°C]
T_i	Injection Temperature [°C]
T_{rate}	Heating Rate [°C/min]
UT	Ultrasonic Testing (Inspection)

This page is intentionally left blank.

1 Introduction

The RTM technique is increasingly applied for the fabrication of thick complex shaped and highly loaded components in aerospace composite structures (wall thickness exceeding 50 mm). Especially the replacement of thick metal forgings of conventional design (of steel or aluminium) is attractive because RTM products may lead to a reduction in weight or cost. This was demonstrated in several technology programmes at the NLR [1-5]. Some examples are given in figure 1.

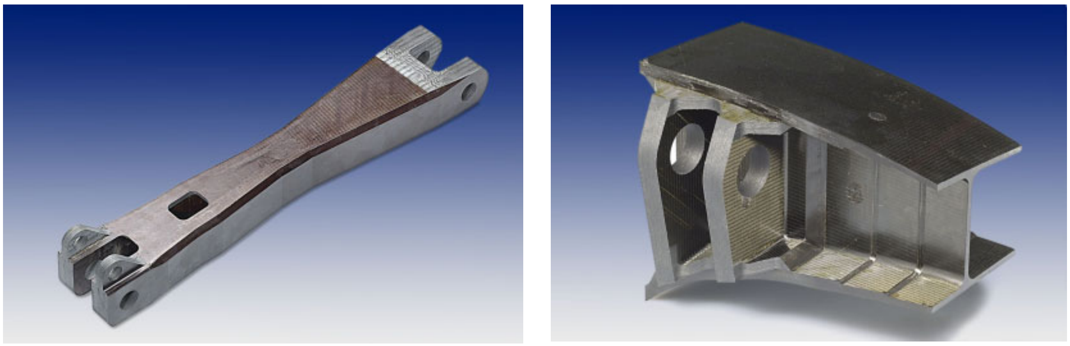


Fig. 1 Composite drag brace and beam with integrated bracket [1-2]

The RTM technique is capable of making components within tight dimensional tolerances and with a high level of part integration; it is even capable of producing products which are impossible to make with the conventional autoclave method for the fabrication of composite structures. Higher volumes of RTM material, however, may decrease the product performance due to cure related material degradation like the formation of shrinkage defects and porosity. For the fabrication of good quality thick RTM products there is hence a need for non-destructive inspection (NDI) techniques for quality control and to optimise the process window for RTM.

The RTM fabrication concept is based on the injection of resin into a mould cavity containing dry fibres (preform). During the injection process at elevated temperature and under pressure, air in the mould is replaced by resin and the fibres are impregnated. Entrapped air and excessive resin can escape through ventilating point(s) at the end of the mould. The product is then cured at a specific temperature and pressure. After curing the product is partly cooled down inside the mould, removed from the mould, and further left to cool down to room temperature. A critical phase in the fabrication process is the curing and cooling down period. Namely, thermal cracks can occur due to polymerisation shrinkage during the cure and due to linear shrinkage during cooling down, especially with thick RTM products (shrinkage percentages of 1 – 2 % by volume are possible). Small micro-cracks within a fibre package but, also, larger shrinkage cracks between the fibre packages can occur. Another defect occurrence inherent with RTM processing is porosity as a result of improper impregnation. This can easily occur with thick RTM products and using a resin of high viscosity. It is therefore essential to select proper resin systems and to optimise the RTM processing steps in order to minimise the risk of cure related defects.

NDI techniques can be applied to monitor the occurrence of cure related defects. It would be very useful to monitor already during the cure step for possible material degradation. The UT technique has been applied for that purpose, using high temperature UT sensors embedded into the mould and working in a transmission mode [6]. However, in the present investigation it was decided to leave the mould intact and to evaluate the feasibility of the AE technique. The AE technique was applied for defect monitoring during the cooling down phase of the RTM product outside the mould to room temperature (although for some products also the cure step and the cooling down phase inside the mould was monitored). Furthermore, the UT C-scan technique was used to assess the final material quality of the RTM product.

2 Experimental

2.1 Specimens

A range of materials was used for the fabrication of the RTM products used in this investigation. A specification of the products is given in [7]. The main material parameters varied were:

- Fabric system: carbon fabric or non-crimp fabric (NCF). The carbon fibre type was HTS Aerospace grade.
- Resin/binder system: varying in order of brittleness from a brittle resin (Cycom 875 RTM with $K_{IC} = 0.5$ MPa.m^{½}), RTM 6, Bakelite EPS 601, Cycom 977-20 RTM, to a tough resin (Cycom 823 RTM with $K_{IC} = 1.6$ MPa.m^{½}). A measure for the brittleness is the fracture toughness K_{IC} or strain energy release rate G_{IC}. Different binder powders to match were used such as Cycom 790, DX 69, Bakelite EPR 05311 and Cytec 7720.
- Specimen dimensions: 200 mm diameter discs with thicknesses of 25, 50 and 90 mm, and rectangular plates with thickness of 28 mm.

Figure 2 gives an example of one of the 90 mm thick RTM discs.

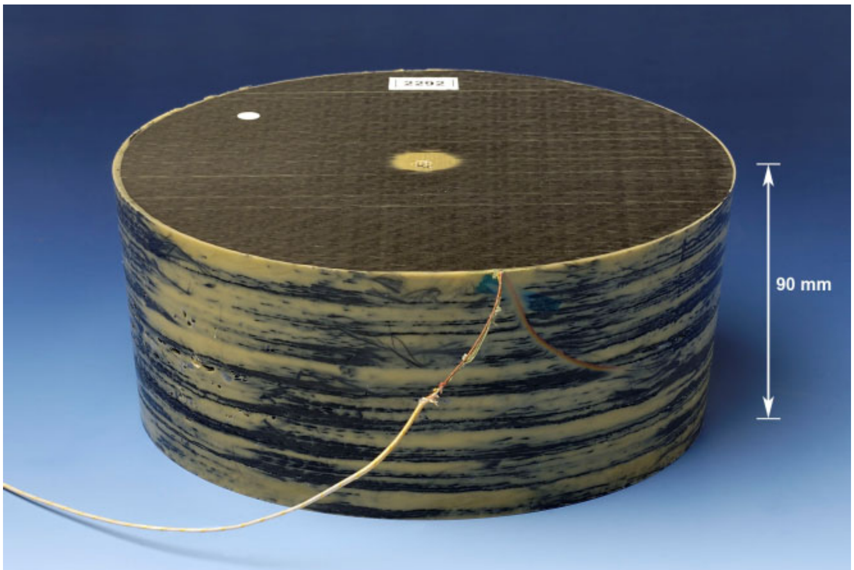


Fig. 2 RTM disc 2292 (Bakelite EPS 601 resin), diameter 200 mm and thickness 90 mm

2.2 RTM processing

The principle and application of the RTM process is well described in [1-5]. An overview of the set-up for RTM processing of 200 mm diameter discs is given in figure 3.

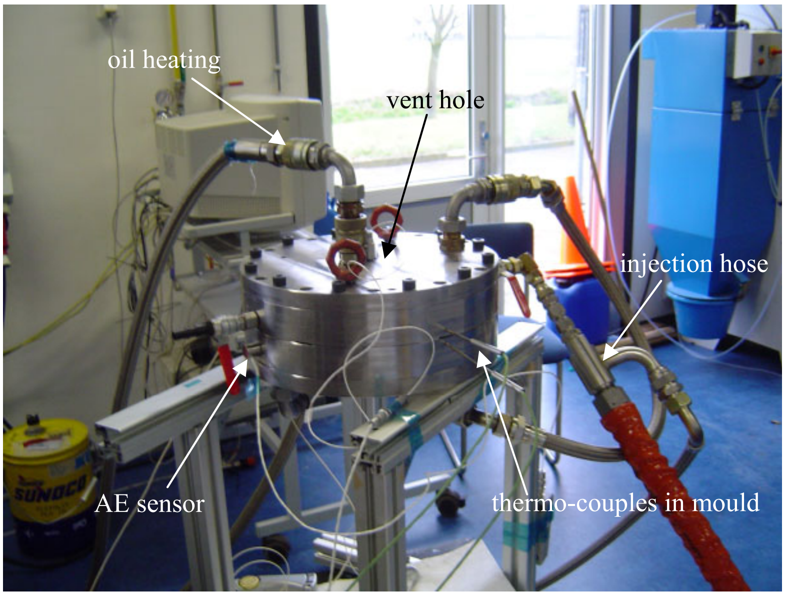


Fig. 3 Overview of set-up for RTM processing of discs

Different mould geometries were used for the fabrication of the RTM discs of different thickness and 28 mm thick rectangular plates. The moulds were somewhat conical shaped to facilitate the demoulding of the cured products. A thermocouple was placed in the centre of the product to monitor the heat development during the production phase. Mould filling occurred by ring injection (preform size somewhat smaller than the mould size).

Examples of basic cure cycles for two resin injection systems (RTM 6 and Bakelite EPS 601) are given in figure 4. During the RTM processing of the products the following parameters were recorded: prescribed temperature-time cycle, temperature distribution of the mould, temperature in the centre of the product, injection pressure and the resin weight decrease in the storage vessel.

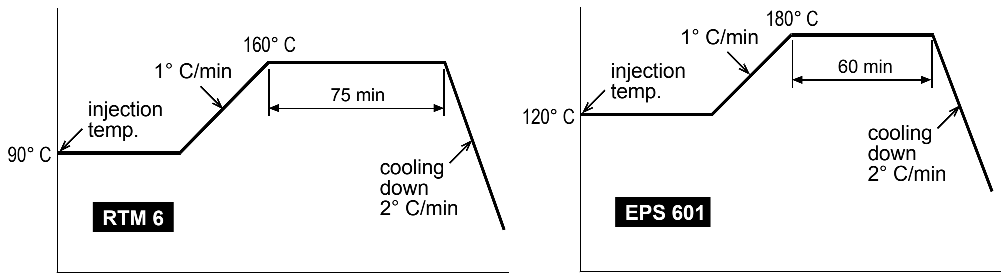
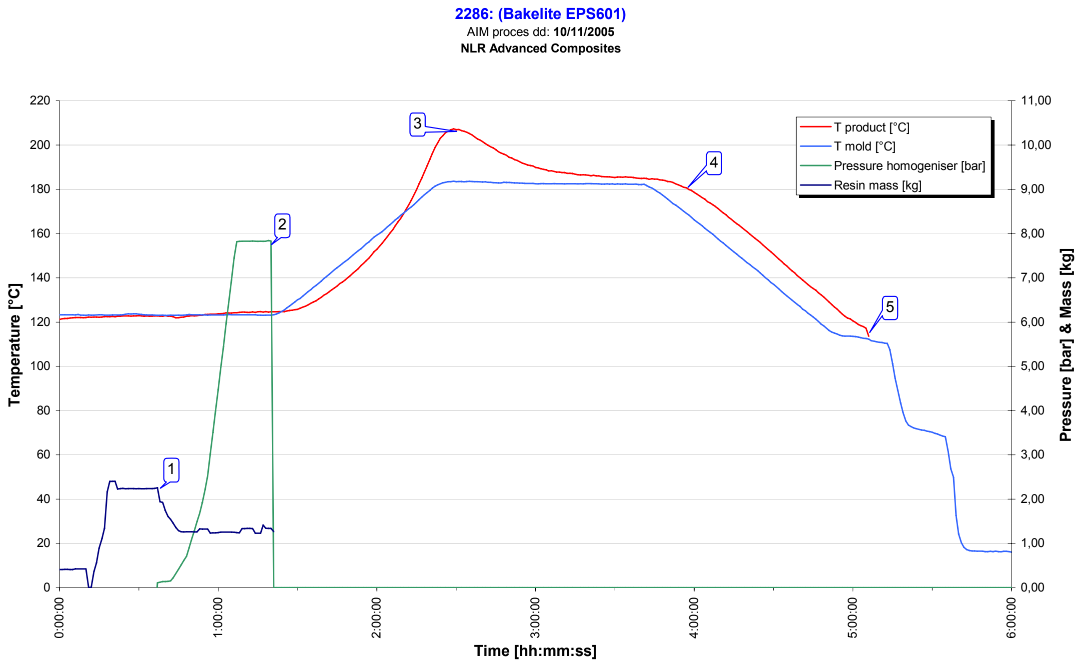


Fig. 4 Basic cure cycles for two resin injection systems [5]

An actual cure cycle for RTM disc 2286 with the Bakelite EPS 601 resin is given in figure 5, with a marking of the most important processing events. The figure illustrates the occurrence of an exothermal peak of heat generation in the centre of the product (3) as a result of polymerisation during the cure phase. Such a peak is unwanted because local overheating can lead to material degradation and unequal curing through the thickness of the product. An effective way to reduce the exothermal peak temperature in the core is the introduction of a dwell time in the cure cycle [5].



1: start of injection, the weight of the resin in the homogeniser drops, 2: end of injection, the injection points are closed, 3: exothermal peak as a result of polymerisation, 4: cure time, 5: demoulding of the product

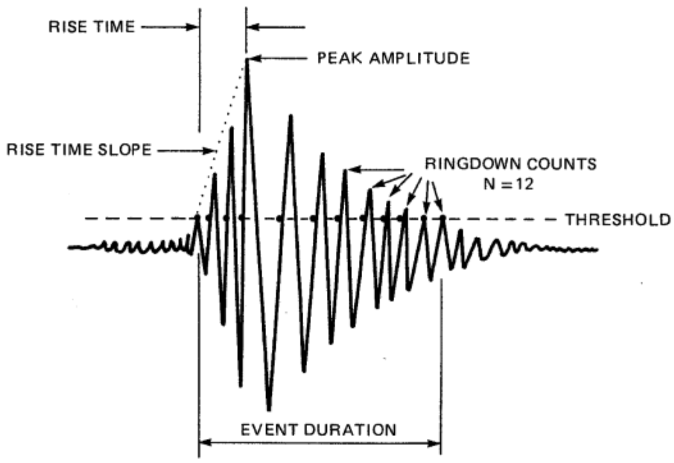
Fig. 5 Actual cure cycle for RTM disc 2286 with the Bakelite EPS 601 resin [4]

An overview of the RTM processing variables used for the different RTM products in this investigation is given in [7]. The main processing parameters varied were:

- Mould wall: fixed or a special design.
- Number of vent holes: 1 or 2 (in the centre of the product).
- Injection temperature.
- Viscosity of the resin at the injection temperature: varying from low (about 35 mPa.s) to high (more than 100 mPa.s).
- Heating rate: 0.5, 1.0 or 2.0 °C/minute.
- Dwell: applied to some products.
- Cure temperature, pressure and time: depending on the resin system.
- Reheat and/or post-cure treatments: applied to some products.

2.3 Acoustic emission

The AE technique is based on the principle that acoustic emissions are generated when defects initiate or grow in a material under stress. Acoustic emission can be defined as the generation of high-frequency transient elastic waves by the rapid release of strain energy from a localised source within a material under stress. When these AE signals, in fact mechanical wave packets, propagate to the surface of the test part, surface waves are created which are finally detected (hit) by special sensors attached to the surface. To provide an adequate acoustic coupling between sensor and material surface, a coupling medium has to be used. AE activity is generally expressed by the number of AE hits per unit of time. An AE hit can be characterised by a number of parameters such as the number of ringdown counts (threshold crossings), peak amplitude, rise time, event duration and energy content (Fig. 6). The energy is the sum of the voltage amplitudes (with a threshold of 0.3 V) of time sections of 10 μs over the AE hit duration. The unit of energy is Volt.sec but, generally, the energy is plotted in arbitrary units.



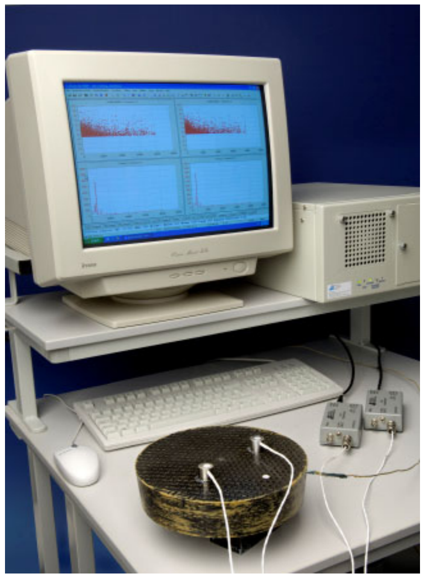


Fig. 6 Waveform characteristics of an AE hit (left) and AE test set-up with two transducers placed on a 50 mm thick RTM disc (right)

The AE technique was applied for defect monitoring during the cooling down phase of the RTM product to room temperature. Mainly the cooling down phase outside the mould was used to compare the AE activity of the different RTM products. For this purpose the RTM products were placed in a vibration-free environment to enable undisturbed measurements. AE inspections were carried out with a four-channel DiSP system of Physical Acoustics Corporation. The AE test set-up is shown in figure 6 (right).

For most experiments only one channel was used for AE recording, employing a 150 kHz resonant transducer R15D with a separate PAC 2/4/6 preamplifier. Only for selected experiments two channels were used, viz. the 150 kHz resonant transducer and a wideband 0-800 kHz sensor WD. The sensors were attached to the surface with Elastosil E43 silicone rubber (Wacker Chemie GmbH), which also functioned as coupling medium. Calibration of inspection was performed by checking the consistency of AE activity from lead-pencil breaks. For all experiments a threshold of 35 dB was used for recording AE hits.

AE monitoring was done during the cooling down phase but the applied time frame (after demoulding) varied for the different products from about 20 hours (disc 2134) to a maximum of more than 10 days (disc 2286). The AE activity can be characterised by a large number of parameters (Fig. 6, left) but the following selection was used for this investigation: time histories (hits, amplitude, energy and temperature versus time), histogram (counts versus amplitude) and correlation plots (amplitude versus energy, duration versus amplitude). These plots were made of the total test duration and at intermediate time intervals of 10000, 20000, 50000, 100000 and 200000 seconds (if measured). At all available time intervals the number of hits, the released energy and the average energy per hit were determined. For overview purposes mostly the AE plots at time intervals of 20000 seconds (approximately the effective cooling down time of 5 to 6 hours to room temperature for a 90 mm thick RTM product) will be given in this paper.

2.4 Ultrasonic inspection

Ultrasonic inspection is a primary technique for the quality control of composite specimens. The UT technique makes use of high-frequency ultrasonic waves, in fact propagating mechanical vibrations with a frequency in the range of about 1-50 MHz. Because air is not an adequate transmitting medium for ultrasonic waves, a coupling medium is generally used between the transducer and material. This can be realised in different ways; for manufacture inspection UT is often carried out with the part totally immersed in water or with the water jet method where the ultrasonic beam is collimated in a narrow water beam.

When an ultrasonic beam is directed onto a material surface, both reflection and transmission of the waves will occur at the material interfaces. The ratio of the reflected and transmitted parts depends on the angle of beam-incidence and on the difference in acoustic impedance (product of material density and wave velocity). Material defects constitute extra interfaces and these will result in extra reflection signals and in a decrease of the transmitted signal. Different pulse-echo or transmission inspection methods can be applied but in this investigation the reflector plate method was used, in fact a double transmission technique (Fig. 7).

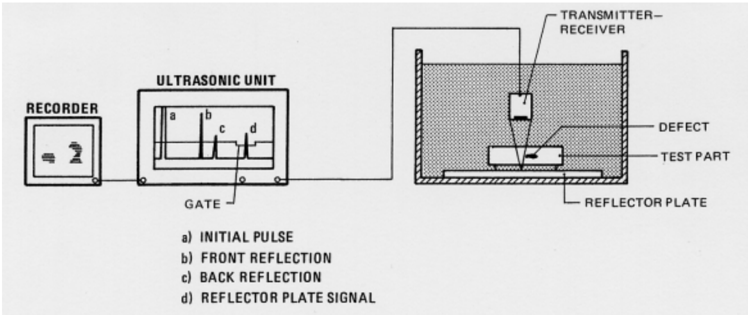


Fig. 7 Ultrasonic C-scan inspection with the immersion technique and reflector plate method

The UT technique was used to assess the final material quality of the RTM products. Inspections were carried out using C-scan equipment AI 1512-S2-T of Automatisation Internationale and data acquisition and analysis equipment of Ultrasonic Sciences Ltd. (Figure 8). Two UT instruments were used, viz. a Sonic-138 (Staveley Instruments, Inc.) or a USIP 40 (Krautkrämer GmbH). Depending on the thickness of the RTM product a 5 MHz (Imasonic IM-5-19-F76) or a 2.25 MHz focused transducer (Imasonic IM-2.25-19-F76) was used. Besides the C-scan presentation also the gain necessary to obtain an 80% screen height on the UT instrument was recorded. This gain value is a measure for the attenuation of ultrasound and, consequently, for the presence of defects in the product.

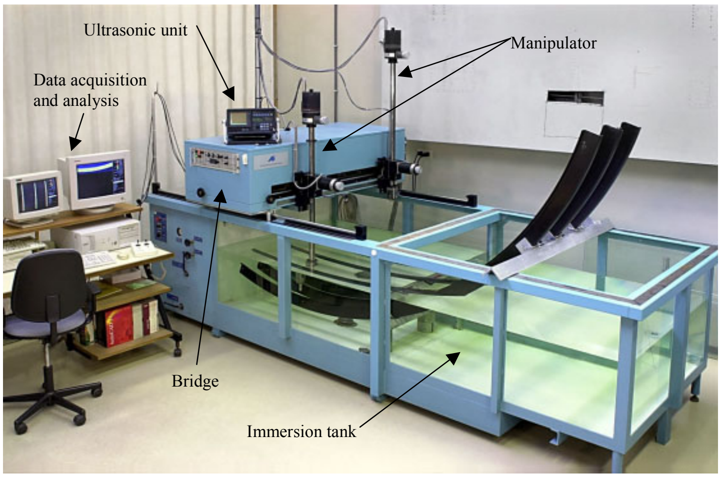


Fig. 8 NLR ultrasonic C-scan equipment

3 Inspection results

3.1 General

Full experimental details and inspection results of the investigation are given in [7]. An overview of the experimental details relevant for the discussion in this paper is given in table 1.

Table 1. Experimental details of the RTM products

Experimental parameters	RTM product 2130	2134	2143	2146	2149	2275	2286	2292	2303
Material									
- fabric	NCF	NCF	NCF	NCF	NCF	fabric	fabric	NCF	fabric
- resin	A	B	A	A	A	C	C	C	D
- resin K_{IC} [MPa.m^{½}]	1.6	0.5	1.6	1.6	1.6	0.9	0.9	0.9	1.36
Mould									
- wall design	special	special	fixed	fixed	special	fixed	fixed	fixed	fixed
- vent hole no.	1	1	2	2	1	1	2	2	2
RTM process									
- T_i [°C]	60	70	60	60	60	120	120	110	90
- η [mPa.s]	35	120	35	35	35	40	40	65	100
- T_{rate} [°C/min]	1	1	1	0.5	1	1	1	1	2
- dwell	--	--	--	--	--	--	--	yes	yes
- T_c [°C]	125	125	120	120	120	180	180	180	180
- T_c time [min]	60	60	60	60	60	60	60	120	185
RTM product									
- size [mm]	600 x 350		Ø 200			Ø 200		Ø 200	
- thickn. [mm]	28		50			50		90	
UT C-scan									
- freq. [MHz]	5	5	5	5	5	2.25	2.25	2.25	2.25
- gain [dB]	47.4	53.0	70.0	?	65.6	58.0	60.0	75.0	75.0
- C-scan quality	good	--	bad	poor	fair	fair	good + del	fair	poor
Cross-section appearance	almost void-free		micro-cracks	shrink cracks, low void %	high void % in area opposite vent hole	low void % in area opposite vent hole	shrink crack, low void %	<< 1% voids, many micro-pores	< 1% voids, no micro-pores

K_{IC} (fracture toughness, RT dry), RT (room temperature), T_i (injection temperature), η (viscosity at T_i), T_{rate} (heating rate), T_c (curing temperature), UT (ultrasonic inspection), NCF (Non Crimp Fabric), resin A (Cycom 823 RTM), resin B (Cycom 875 RTM), resin C (Bakelite EPS 601), resin D (Cycom 977-20) RTM, del (delamination), -- (not performed)

An overview of the AE results during the cooling down phase outside the mould for the different RTM products is given in table 2.

Table 2. AE results for the RTM specimens (hits, energy, average energy per hit). Cooling down phase of the RTM products outside the mould

Specimen and AE activity		Time Interval [ksec] 0 - 10	10 - 20	20 - 50	50 - 100	100 - 200	> 200	Total	Total time [s]
2130	Hits	318	19	10	6	--	--	353	73,815
	Energy	469	7	2	2	--	--	480	
	Average energy	1.5	0.4	0.2	0.3	--	--	1.4	
2134	Hits	2232	326	114	53	--	--	2725	77,590
	Energy	65224	18497	1107	209	--	--	85037	
	Average energy	29.2	56.7	9.7	3.9	--	--	31.2	
2143	Hits	32489	13088	14962	2670*	5117*	1213	69539	234,462*
	Energy	133395	75537	86957	15901	19216	7254	338260	
	Average energy	4.1	5.8	5.8	6.0	3.8	6.0	4.9	

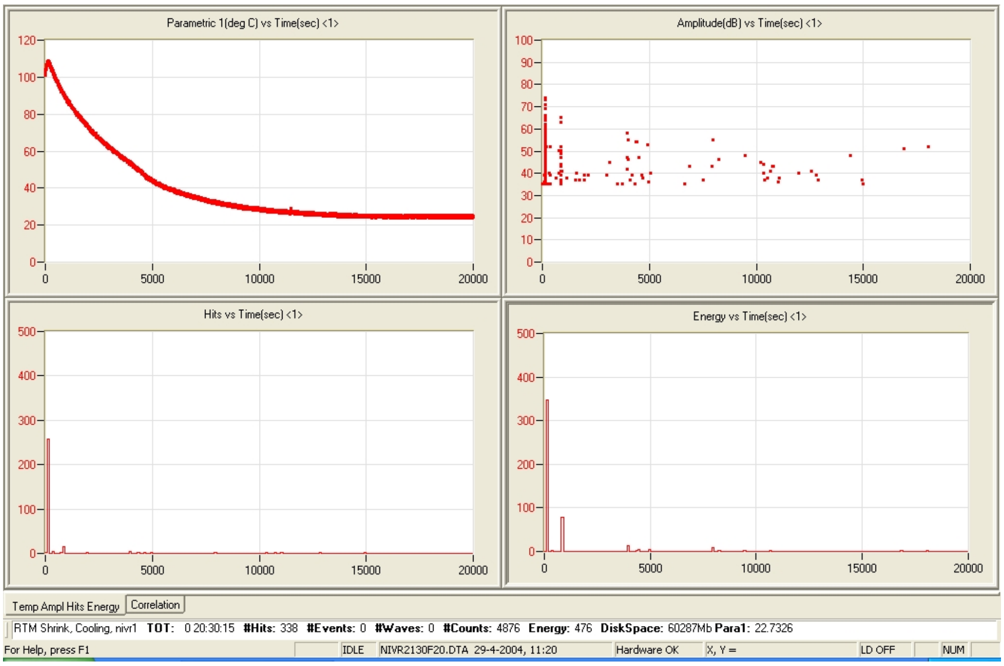
2146	Hits	9469	1658	1583	2921	1845	--	17476	170,454
	Energy	35265	9852	13813	15191	8585	--	82706	
	Average energy	3.7	5.9	8.7	5.2	4.7	--	4.7	
2149	Hits	1537	414	392	246	--	--	2589	81,032
	Energy	21309	1887	1954	1454	--	--	26604	
	Average energy	13.9	4.6	5.0	5.9	--	--	10.3	
2275	Hits	9230	1132	834	323	--	--	11519	80,303
	Energy	11079	1870	1616	955	--	--	15520	
	Average energy	0.8	1.7	1.9	3.0	--	--	1.0	
2286	Hits	8931	1085	1077	494	612	1292	13491	945,259
	Energy	12640	2862	2452	1090	1337	2846	23227	
	Average energy	1.4	2.6	2.3	2.2	2.2	2.2	1.7	
2292	Hits	4337	1755	1026	379	188	407	8092	571,438
	Energy	14621	5702	6050	353	176	293	27195	
	Average energy	3.4	3.2	5.9	0.9	0.9	0.7	3.4	
2303	Hits	526	191	59	19	10	19	824	490,388
	Energy	24569	11179	507	2	84	43	36384	
	Average energy	46.7	58.5	8.6	0.1	8.4	2.3	44.2	

* intermittent measurements, -- no measurements

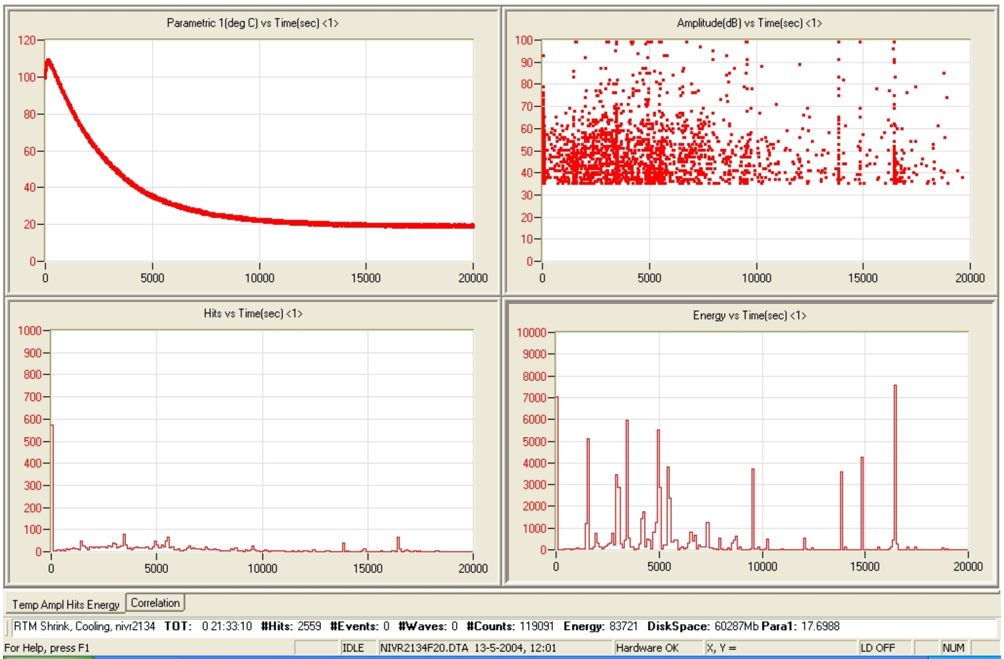
In the following sections a selection of the inspection results is presented.

3.2 Effect of resin system

The AE results of 28 mm thick RTM plates 2130 and 2134 are summarised in figure 9 (first 20000 seconds of cooling down to room temperature). The figure illustrates the effect of *resin system* on the AE activity. Plate 2130 was made using carbon NCF and Cycom 823 RTM resin which is relatively tough considering its fracture toughness K_{IC} of 1.6 MPa.m^{½} , while plate 2134 was made using the same reinforcement but with Cycom 875 RTM resin which is relatively brittle (K_{IC} = 0.5 MPa.m^{½}). Both plates received the same cure treatment using the same special mould design (with 1 vent hole) but the difference in AE activity is striking. The more brittle Cycom 875 RTM resin system (plate 2134) developed a more than 7 times higher number of hits and even a more than 175 times higher AE energy level than the tougher Cycom 823 RTM resin system. This difference can probably be explained by the generation of more micro-cracks in the brittle resin system. The ultrasonic gain measurements (table 1) show a 6 dB higher gain level for plate 2134 which is also an indication of higher damage accumulation.



337 AE hits, released energy 476 Plate 2130 with tough Cycom 823 RTM resin

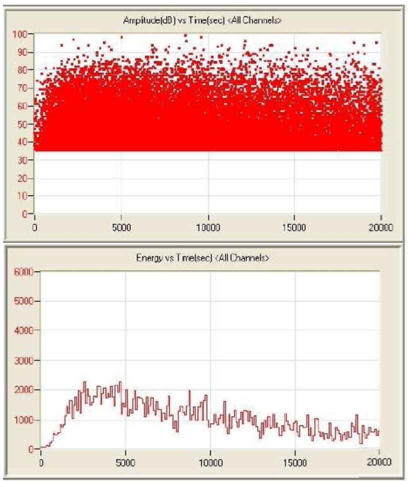


2558 AE hits, released energy 83721 Plate 2134 with brittle Cycom 875 RTM resin

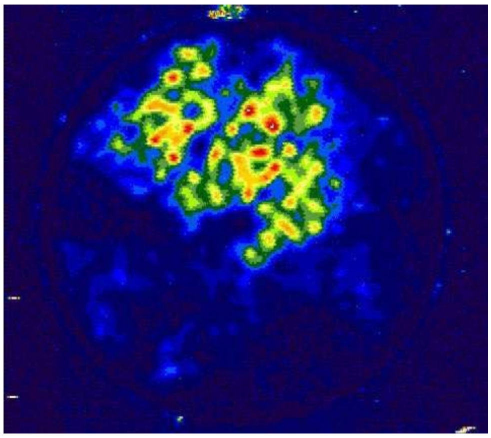
Fig. 9 AE time histories during the first 20000 seconds of cooling down for 28 mm thick RTM plates 2130 (upper figure) and 2134 (lower figure)

3.3 Effect of heating rate and mould design

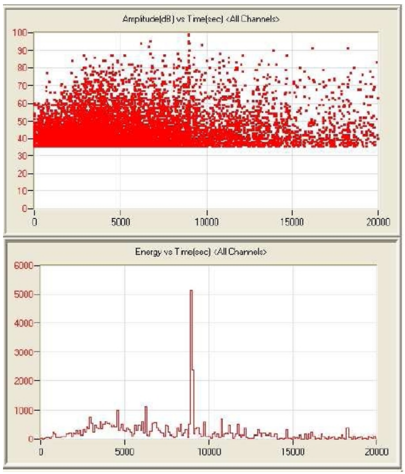
The AE results (first 20000 seconds of cooling down) and C-scan results of 50 mm thick RTM discs 2143, 2146 and 2149 are summarised in figure 10. Cross-sections of discs 2146 and 2149 are given in figure 11. Figure 10 illustrates the effect of *heating rate* (from the injection temperature to the final cure temperature) on the AE activity during cooling down. Discs 2143 and 2146 were made using carbon NCF and Cycom 823 RTM resin (Cytec), but for disc 2143 a standard heating rate of 1 °C/min and for disc 2146 a relatively low heating rate of 0.5 °C/min was used. Figure 10 shows that the number of AE hits and generated energy level is obviously smaller for a lower heating rate indicating a lower level of damage accumulation. The C-scan presentations indeed indicate a somewhat better material quality for disc 2146, although the attenuation is very high especially in the centre and some outer parts. A cross-section of disc 2146 is given in figure 11 (left) showing resin-rich areas and large shrinkage cracks in the middle fibre layers of the laminate. The locations of the shrinkage cracks in the cross-section coincide with the areas of high attenuation in the C-scan. The shrinkage cracks are probably associated with the high energy peak occurring at about 9000 seconds. A further remarkable observation is the large number of AE hits that continue to occur well after the discs have cooled down (especially for disc 2143).



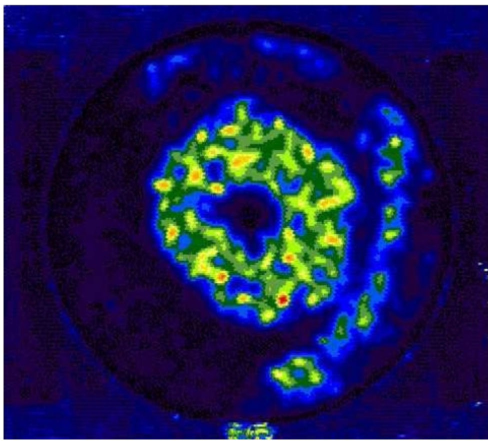
45577 AE hits, released energy 208932



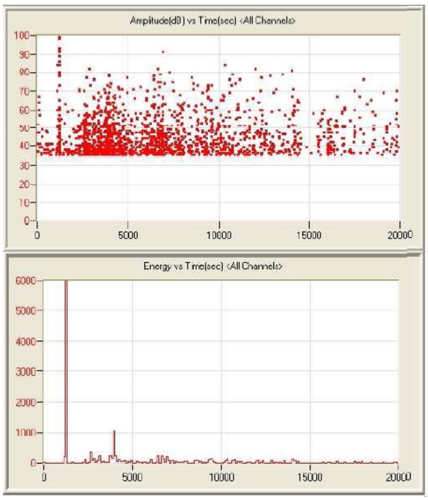
C-scan of disc 2143, heating rate 1 °C/min, 'hard' mould with 2 vent holes. Resin-rich areas and small shrinkage cracks



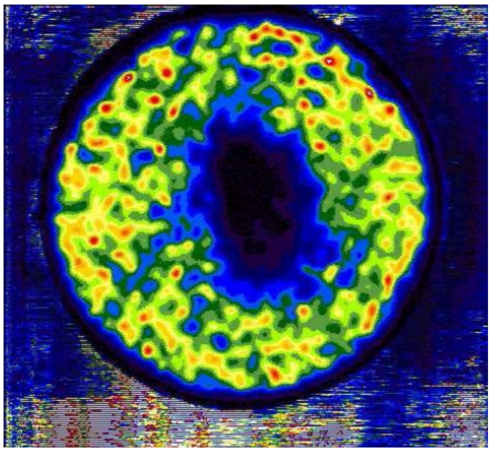
11127 AE hits, released energy 45117



C-scan of disc 2146, heating rate 0.5 °C/min, 'hard' mould with 2 vent holes. Resin-rich areas and shrinkage cracks

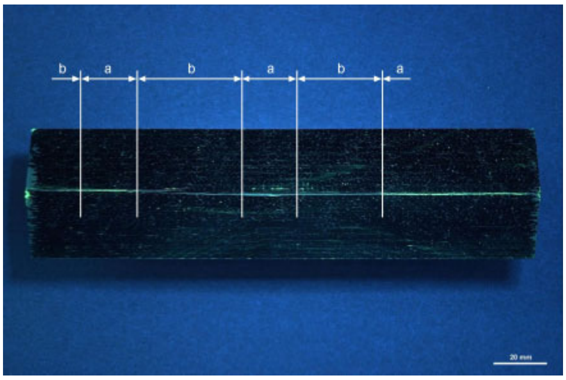


1951 AE hits, released energy 23196

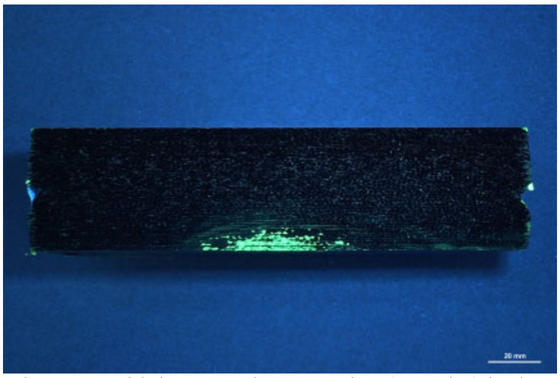


C-scan of disc 2149, heating rate 1 °C/min, special mould with 1 vent hole. High porosity % in the centre, opposite the vent hole

Fig. 10 AE time histories during the first 20000 seconds of cooling down (amplitude, energy) and UT C-scans for 50 mm thick RTM discs 2143, 2146 and 2149 (Cycom 823 RTM resin)



Disc 2146 with shrinkage cracks (a) and resin rich areas (b)



Disc 2149 with improper impregnation (porosity) in the central product region opposite the vent hole location

Fig. 11 Cross-sections of RTM discs 2146 and 2149 (Cycom 823 RTM resin)

Figure 10 also shows that the AE activity is further reduced significantly by application of a *mould of special design* (with 1 vent hole) instead of a fixed wall (with 2 vent holes), compare disc 2149 with 2143 and 2146. This is illustrated by both the amplitude and energy distributions. Remarkable is the high energy peak (and amplitude exceeding 99 dB!) occurring for disc 2149 at about 1210 seconds. The C-scan presentation of disc 2149 shows that the material quality of the disc is much better than discs 2143 and 2146, although the attenuation is again very high in the centre of the product. A cross-section of the disc (Fig. 11, right) shows improper impregnation (porosity) in the central product region opposite the single vent hole location. The location of the porosity in the cross-section coincides with the area of high attenuation in the C-scan. The porosity area occurs because once the mould has been filled there is hardly any flow possible under continued injection pressure to remove remaining air bubbles. This was illustrated using the computer code RTM-worx (Company Polyworx B.V.) by calculating the steady state velocity in the RTM mould for different thicknesses, see figure 12 [5].

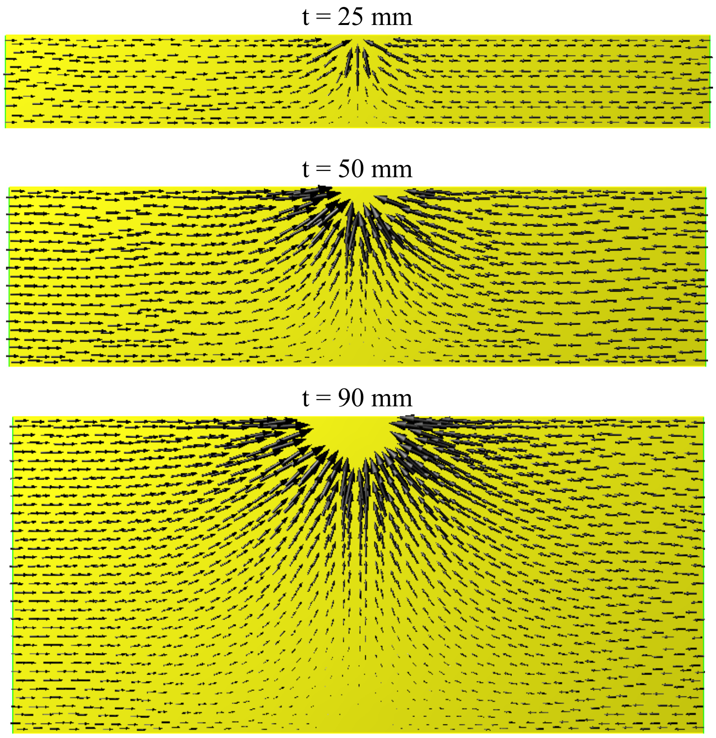


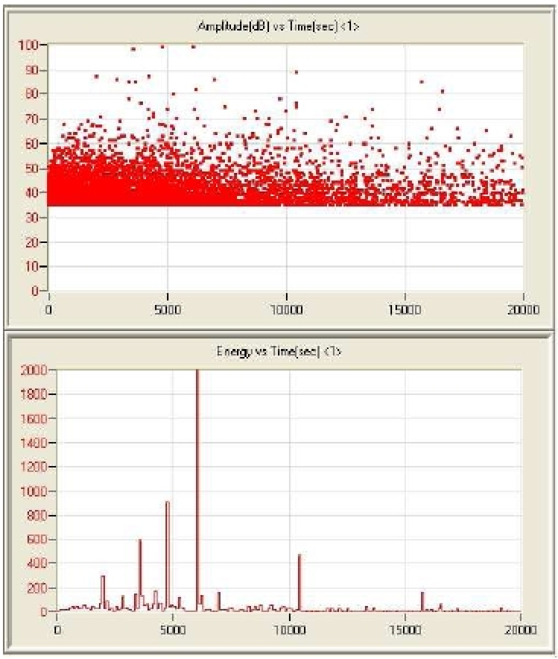
Fig. 12 Steady state velocity in an RTM mould with 1 vent hole, for different thicknesses (t) of the mould [5] (Conditions: ring injection ΔP = 1 bar, $K_{isotropic} = 10^{-10}$ m^2, viscosity = 0.01 Pa.s)

It is finally noted that the results in figures 10 and 11 suggest a relation between AE activity, UT attenuation level and the presence of cure related defects.

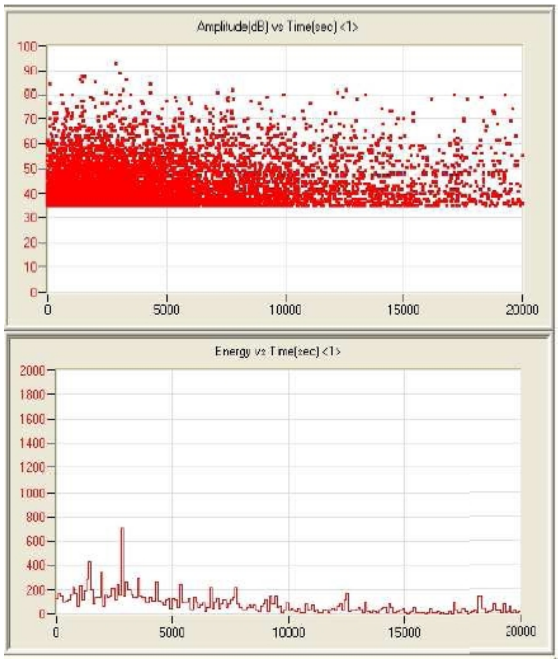
3.4 Other measurements on thick RTM products

The AE results (first 20000 seconds of cooling down), C-scan results and cross-sections of 50 mm thick RTM discs 2275 and 2286 are summarised in figure 13. Both discs were made using a carbon fabric and Bakelite EPS 601 resin (Hexion). This resin is a 180 °C curing system and medium brittle/tough considering its fracture toughness of 0.9 $MPa.m^{½}$. Both discs were manufactured with a mould with fixed wall but the number of vent holes was one for disc 2275 and two for disc 2286.

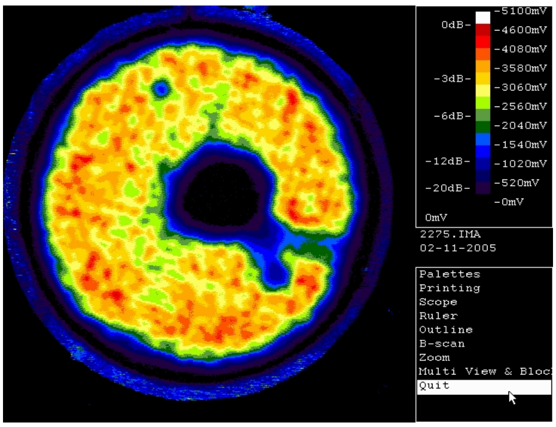
Figure 13 shows that the average AE energy level is somewhat higher for disc 2286 while for disc 2275 most released energy is associated with a few high-energy peaks (and with amplitudes exceeding 99 dB). For both discs a significant number of hits continue to occur well after the discs have cooled down. The C-scans look similar for the two discs (the material quality of disc 2286 is somewhat better, see also the UT gain levels in Table 1) but the cross-sections show that the origins of the central product regions with high UT attenuation are very different. The high attenuation for disc 2275 is caused by porosity in the region opposite the vent hole location (mould with only 1 vent hole), while the high attenuation for disc 2286 (mould with 2 vent holes) is caused by a large thermal crack. It is expected that such a large thermal crack is associated with high energy AE peaks but that has not been demonstrated for disc 2286. Probably, the large crack in disc 2286 had already initiated before the AE monitoring was started. The cross-sections of the two discs further demonstrate that the use of a mould with two vent holes decreases the porosity content significantly.



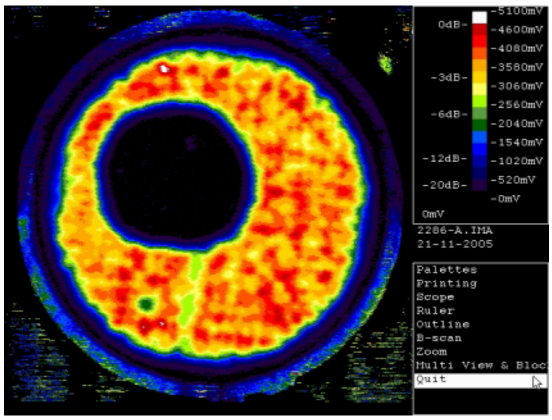
10362 AE hits, released energy 12949



10016 AE hits, released energy 15502



Ultrasonic C-scan of disc 2275



Ultrasonic C-scan of disc 2286

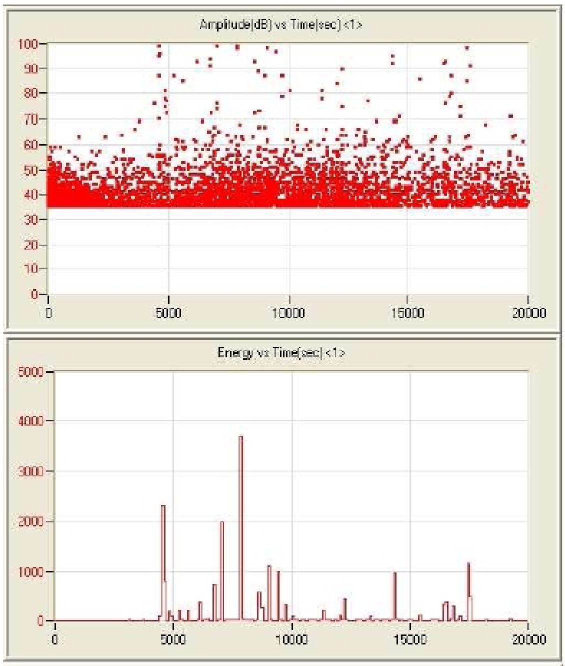
Cross-section of disc 2275: porosity in the central product region opposite the vent hole location (mould with 1 vent hole)

Cross-section of disc 2286: some porosity and a large thermal crack (mould with 2 vent holes)

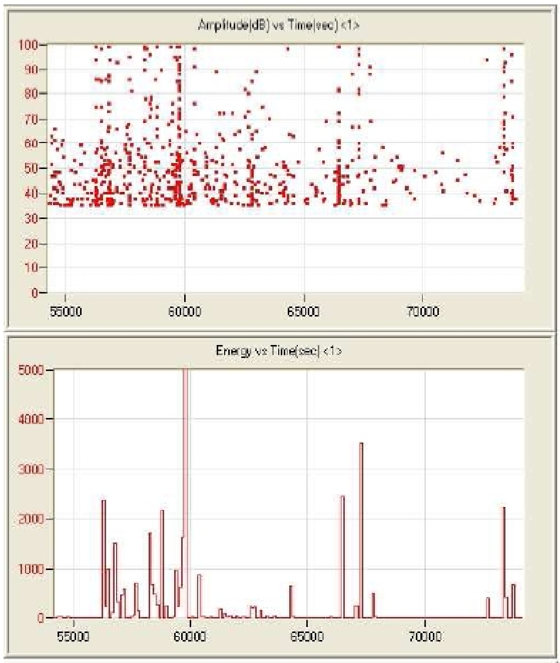
Fig. 13 AE time histories during the first 20000 seconds of cooling down (amplitude, energy), UT C-scans and cross-sections of 50 mm thick RTM discs 2275 (left) and 2286 (right)

The AE results (first 20000 seconds of cooling down), C-scan results and cross-sections of 90 mm thick RTM discs 2292 and 2303 are summarised in figure 14. Disc 2292 (Fig. 2) was made using carbon NCF and Bakelite EPS 601 resin (Hexion), and disc 2303 using a carbon Priform fabric (with interweaved thermoplastic thread) and Cycom 977-20 RTM resin (Cytec).

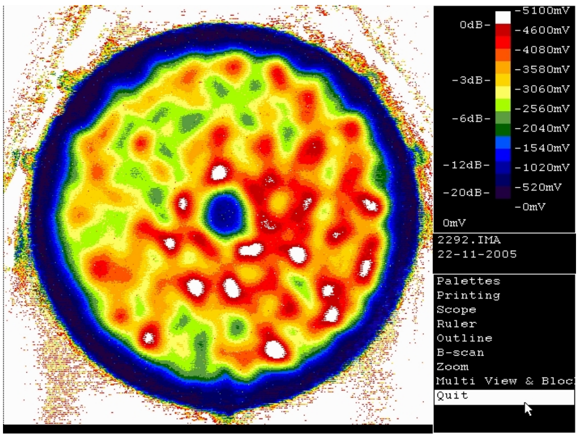
Both resin types are 180 °C curing systems. Bakelite EPS 601 resin is medium brittle/tough and Cycom 977-20 RTM resin is relatively tough considering the fracture toughness's of 0.9 and 1.4 MPa.m^{½}, respectively. Both discs were manufactured with a mould with fixed wall and two vent holes. For both discs a dwell period was included in the RTM processing (for disc 2303 especially to dissolve the thermoplastic thread inside the fabric).



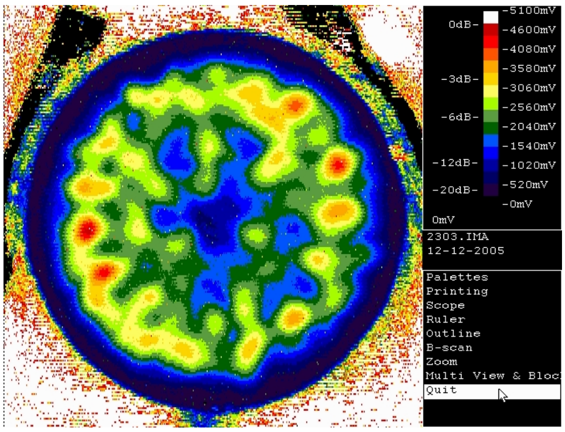
6092 AE hits, released energy 20323



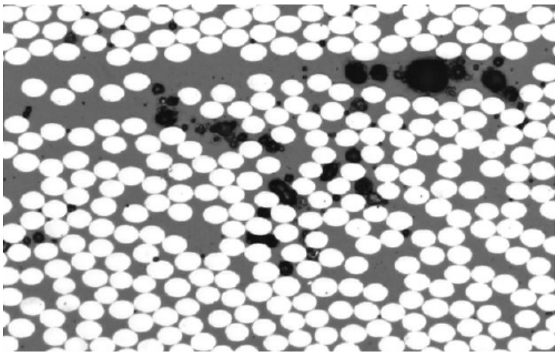
717 AE hits, released energy 35748



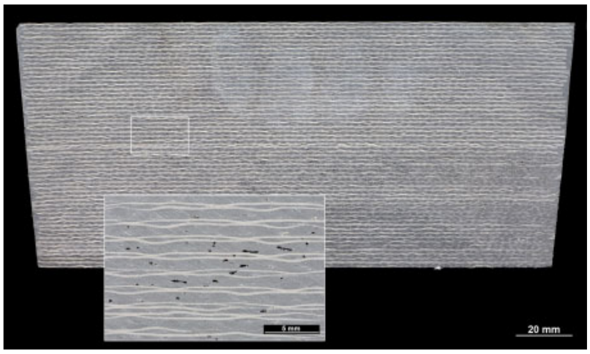
Ultrasonic C-scan of disc 2292



Ultrasonic C-scan of disc 2303



Cross-section area of RTM disc 2292: very low amount of porosity (<< 1%) distributed randomly, but many micro-pores (with diameter in the range of the fibre diameter ~ 19 μm)



Cross-section of RTM disc 2303: low amount of porosity (< 1%) concentrated in the middle 30 layers of the laminate, but no micro-pores

Fig. 14 AE time histories during the first 20000 seconds of cooling down (amplitude, energy), UT C-scans and cross-sections of 90 mm thick RTM discs 2292 with Bakelite EPS 601 resin (left) and disc 2303 with the tougher Cycom 977-20 RTM resin (right)

Figure 14 (and Table 2) shows that the AE activity in terms of number of AE hits for disc 2292 is about 10 times higher than for disc 2303 but the total amount of released AE energy is higher with disc 2303. The material quality of disc 2303 is also lower than for disc 2292 (in terms of C-scan appearance and porosity content). The higher degree of porosity in disc 2303 is probably associated with the higher viscosity of the Cycom 977-20 RTM resin at the time of injection (Table 1). The higher number of AE hits in disc 2292 was in first instance associated with more micro-cracks in the more brittle Bakelite EPS 601 resin system. However, microscopic examination of the cross-section of disc 2292 revealed no micro-cracks at all but, instead, a large number of micro-pores (pores with a size in the range of the fibre diameter of about 19 μm) in spite of the low overall porosity content (<< 1%). In disc 2303 with higher porosity content (still < 1%), on the other hand, no micro-pores (and also no micro-cracks) were observed; the porosity areas in this disc are generally much larger than 10 times the fibre diameter (Fig. 14). Those voids also cause the relative high attenuation in the C-scan of disc 2303. So for these discs it seems that high AE activity in terms of number of AE hits is not associated with micro-cracks but with micro-pores.

Finally it is noted that the RTM process was capable to produce exceptionally thick RTM products without much porosity (< 1%) and without any large shrinkage cracks.

4 Discussion

The inspection results have shown that AE and UT C-scan are useful tools to assess the material quality of RTM products: AE for defect monitoring during the cooling down phase of the products to room temperature, and UT C-scan for the assessment of the final quality of the products. The results also suggest a relation between AE activity, UT attenuation level and the presence of cure related defects in RTM products. A higher level of AE activity (more in terms of energy than number of AE hits) corresponds in general with a higher ultrasonic attenuation level and a lower quality of the final RTM product. The exact role of different cure related defect types such as small micro-cracks, larger shrinkage cracks and porosity content (micro-pores and much larger voids) on the AE activity, however, remains to be established. A contributing factor is that in the present investigation in fact too many RTM processing parameters were varied, often for one product simultaneously. Further investigation with a systematic study of the influence of single RTM processing parameters is therefore recommended. More microscopic examination of defects in cross-sections is also necessary. However, based on the preliminary results of this investigation the following trends for the detection of cure related defects are expected:

- Micro-cracks and micro-pores: result in many AE hits (especially in the lower AE amplitude regime) but are less or not visible in the UT C-scan.
- Larger shrinkage cracks (delaminations): result in AE hits of high energy and are well visible in the UT C-scan.
- Larger porosity areas (voids): result in few AE hits but are well visible in the UT C-scan.

The AE technique was mainly applied for defect monitoring during the cooling down phase to room temperature *after* removal of the RTM products from the mould. This has the advantage that only the AE activity of the RTM product itself is recorded, but has the disadvantage that defects associated with polymerisation shrinkage and first stage linear shrinkage are missed. Only in some cases AE monitoring was already applied during the cure phase and cool down period inside the mould (AE transducer attached to the outer surface of the mould), see for example figure 15 for disc 2303.

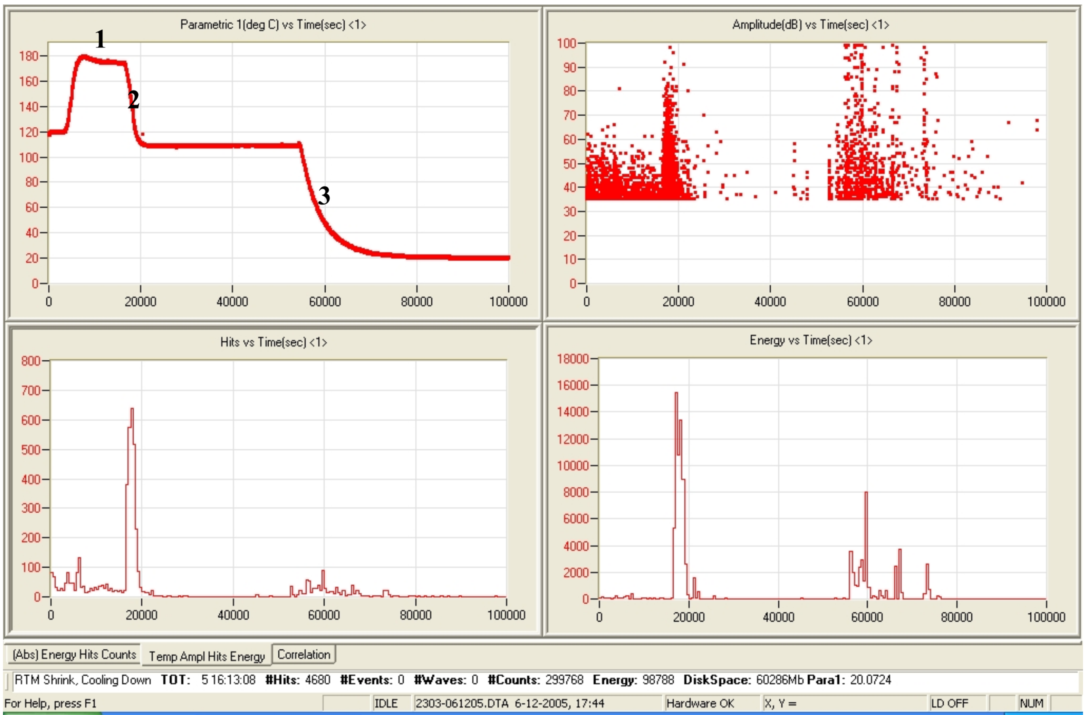


Fig. 15 AE time histories for disc 2303: first 100,000 seconds of total AE monitoring during cure (1), cool down inside mould (2) and cool down outside mould (3). Demoulding of disc at 54240 seconds

Figure 15 shows that during cure a number of AE hits occurs but of relatively low energy. This activity is probably due to polymerisation shrinkage effects. During the cool down period inside the mould a significant number of hits and of very high energy occurs when compared with the AE activity recorded during the cool down period outside the mould. This activity can be caused by real shrinkage defects but can also be associated with the coming apart of the RTM product from the inside of the mould. At this stage it is not yet possible to exactly detail the different AE activities but, at any case, it is recommended to include the RTM processing time inside the mould in future investigations with AE monitoring.

5 Conclusions and recommendations

1. Acoustic emission and ultrasonic C-scan are useful tools to assess the material quality of RTM products: AE for defect monitoring during the cooling down phase of the products to room temperature, and UT C-scan for the assessment of the final material quality of the products.
2. The results of the investigation show a relation between AE activity, UT attenuation level and the presence of cure related defects in RTM products. A higher level of AE activity (in terms of AE energy) corresponds in general with a higher ultrasonic attenuation level and a lower quality of the RTM product.
3. The influence of RTM material and process parameters on the AE activity and final material quality was studied. The following trends were observed:
   - A brittle resin system generally results in higher AE activity in terms of number of AE hits (especially in the lower AE amplitude regime), probably caused by the generation of more micro-cracks (or micro-pores).
   - An RTM mould of special design instead of a fixed wall results in a significant reduction of the AE activity (in terms of number of AE hits and generated energy level) and in a higher material quality (less attenuation in the C-scan presentation). The use of only 1 vent hole in the mould results in a higher porosity content, especially in the central product region opposite the vent hole location.
   - A lower heating rate (from the injection temperature to the curing temperature) results in a reduction of the AE activity and in a somewhat higher material quality.
4. During the investigation too many RTM processing parameters were varied for the products. Therefore, the exact role of different cure related defect types such as small micro-cracks, larger shrinkage cracks and porosity content (micro-pores and much larger voids) on the AE activity remains to be established. Further investigation should also include the AE monitoring of the RTM cure and cool down time inside the mould to study the effects of polymerisation shrinkage and first stage linear shrinkage.

References

[1] Thuis, H.G.S.J.; C*omposite landing gear component for aerospace applications*, paper presented at the 24th International Congress of the Aeronautical Sciences, Yokohama, Japan, 29 August – 3 Sept 2004, report number ICAS-2004-5.3.2..

[2] Thuis, H.G.S.J.; *Developments of composite manufacturing technologies at NLR – Resin Transfer Moulding*, NLR report TP-2005-159, June 2005, paper presented at the Society of Allied Weight Engineers, Annopolis (Maryland), USA, 16-18 May 2005.

[3] Hart, W.G.J. ‘t; Ubels, L.C; *Damage tolerance properties of carbon/epoxy lugs made from Resin Transfer Moulded (RTM) plates*, NLR report TP-2005-356, paper presented at the Advancing with Composites 2005 Conference, Naples, Italy, 11-14 October 2005.

[4] Grouve, W.J.B.; *Characterisation of new RTM resin systems for aerospace applications*, NLR report AVST-2005-123, Amsterdam, December 2005.

[5] Hart, W.G.J. ‘t; *Manufacturing of 50 mm thick carbon/epoxy discs by Resin Transfer Moulding*, NLR report CR-2005-683, Amsterdam, December 2005.

[6] Dessendre, M.; Thevenot, F.; Tretout, H.; *NDT technology to assist regulation system for smart injection*, paper presented at the 8th European Conference on Non-Destructive Testing, Barcelona, Spain, 17-21 June 2002, paper AS-8-1.

[7] Heida, J.H.; *Acoustic emission and ultrasonic inspection for the monitoring of cure related defects in thick RTM products*, NLR report AVGS-2006-036, Amsterdam, May 2006.