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Safety Standards and Inspection Techniques For Natural Gas Composite Cylinders

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	Many new designs of lightweight composite cylinders used in the transportation industry are not covered by existing regulatory standards ISO TO 58/SC 200/C 11 is developing a standard for composite cylinders that con					lers that can
	provide a basis for replacement of th	e CAN/CSA B339 st	andard currently	used by Transi	posite cylinc	Powertech
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	composite cylinder designs used on	compressed natural	gas-fuelled vehi	cles.		
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	enhanced through the development	of a nondestructive i	a in transportation	d. Studies were	conducted	on the ability
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	simplifié pour le contrôle par émi	ssion acoustique de	es bouteilles en	composite. D'a	autres essai	is dans des	
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SUMMARY

High pressure cylinders of composite-wrapped design are utilized for compressed gas as an on-board fuel, and for the transportation of dangerous goods. The application of composite technology to the design and use of lightweight composite cylinders in the transportation industry is growing rapidly. Many applications are not covered by existing regulatory standards. ISO TC 58/SC 3/WG 11 is currently developing a standard for composite cylinders that will likely be adopted by Transport Canada. To ensure the safe development of high pressure composite cylinders in the transportation industry, it was important to apply the experience of the natural gas vehicle industry to the ISO standard. Powertech Labs participated in a critical meeting of ISO TC 58/SC 3, wherein basic requirements for the design of composite cylinders were established. It was recommended that Canada continue its participation on the ISO subcommittee to expedite the development of a composite cylinder standard.

Safety of high pressure cylinders used in transportation applications could be greatly enhanced through the development of a non-destructive inspection method for composite reinforced designs. The ability of acoustic emission (AE) devices to detect impact damage on carbon fibre-wrapped cylinders was studied. The study showed that AE could readily detect composite damage under controlled test conditions, and that a simplified test procedure was possible. Field tests using AE on cylinders pressurized with compressed natural gas were recommended.

SOMMAIRE

Des bouteilles haute pression à frettes en composite sont utilisées pour le stockage de gaz comprimé alimentant les véhicules, et pour le transport de marchandises dangereuses. Le recours aux composites pour la conception de bouteilles légères destinées au secteur des transports connaît un essor rapide. Or, nombre de ces bouteilles échappent aux normes en vigueur. L'ISO TC 58/SC 3/WG 11 travaille présentement à l'élaboration d'une norme sur les bouteilles en composite, qui, selon toute vraisemblance, sera adoptée par Transports Canada. Pour garantir que la percée des bouteilles haute pression en composite dans le secteur des transports ne compromette en rien la sécurité, il était essentiel que l'industrie des véhicules alimentés au gaz naturel soit mise à contribution. C'est ainsi que Powertech Labs a participé à une réunion cruciale de l'ISO TC 58/SC 3, à laquelle ont été établies les spécifications de base pour la conception des bouteilles en composite. À la suite de cette réunion, il a été recommandé que le Canada continue de participer aux travaux du souscomité de l'ISO, de façon que l'on dispose d'une norme au plus tôt.

La mise au point d'une méthode de contrôle non destructive pourrait grandement accroître la sûreté des bouteilles haute pression en composite utilisées pour le transport de gaz comprimés. Une étude a été menée pour déterminer la capacité des appareils à émission acoustique de détecter les dommages subis par des bouteilles à frettes en fibre de carbone à la suite d'un choc. Cette étude s'est avérée concluante, révélant la possibilité de détecter rapidement, par émission acoustique, les dommages subis par le composite lors d'essais contrôlés, et la faisabilité d'un protocole d'essai simplifié. Les chercheurs ont recommandé de soumettre à d'autres essais à émission acoustique, en conditions réelles de service cette fois, des bouteilles remplies de gaz naturel comprimé.

TABLE OF CONTENTS

1. CYLINDER STANDARDS	1
2. DEVELOPMENT OF ISO STANDARD FOR COMPOSITE CYLINDERS	2
3. CYLINDER INSPECTION TECHNIQUES	
4. Research Approach	4
5. TEST METHODS	5
5.1 Overview	5
5.2 Comdyne SCC Tests	
5.3 EDO Impact Tests	6
6. DISCUSSION	7
7. CONCLUSIONS	
8. RECOMMENDATIONS	9
REFERENCES	

APPENDICES

- A Report of the Special Meeting of ISO TC 58/SC 3 Gas Cylinder Design, London, UK March 1999
- B Final Report on Acoustic Emission Testing

1. CYLINDER STANDARDS

The use of high pressure cylinders reinforced with composite wrapping for air breathing, medical and natural gas vehicle applications is increasing due to their lightweight properties. Lightweight high pressure cylinders of composite-reinforced design also have other potential applications, including use in tube trailers for the large-scale transportation of compressed gases and for carrying compressed hydrogen as a vehicle fuel.

Permits issued by Transport Canada under the CAN/CSA B339 standard "Cylinders, Spheres, and Tubes for the Transportation of Dangerous Goods" for the use of compositewrapped cylinders require that the designs be hydrostatically tested every three years. Few facilities have test equipment capable of performing the hydrostatic test on large cylinders. In addition, hydrostatic testing has limited ability to detect metal fatigue and composite stress rupture damage. Hydrostatic testing also requires removal of the cylinder from the vehicle or trailer, a significant expense that increases the risk of damage to cylinders during handling.

For steel tube trailers, Transport Canada has recently issued a permit to Tektrend allowing the use of acoustic emission (AE) as a retest method. While hydraulic or pneumatic pressurization of the tube is still required to perform the AE test, the advantage is that the test can be conducted in situ. To facilitate the economic and safe use of composite-wrapped cylinders on vehicles for use either as fuel storage or for the transportation of dangerous goods, it is necessary to develop a similar non-destructive inspection method for in situ inspection of the composite wrap.

2. DEVELOPMENT OF ISO STANDARD FOR COMPOSITE CYLINDERS

Composite cylinders used for the storage or conveyance of compressed or liquefied gases are in greater demand as the need for lightweight cylinders increases in the marketplace. The International Standards Organization under ISO TC58 SC3 Working Group 11 is preparing an international standard (ISO CD 11119) to provide a specification for the design, manufacture, inspection and testing of composite cylinders for worldwide usage. The objective is to balance design and economic efficiency against international acceptance and utility.

The standard aims to eliminate concern about climate, duplicate inspection and restrictions currently existing because of lack of definitive international standards. In Canada, CAN/CSA B339 "Cylinders, Spheres, and Tubes for the Transportation of Dangerous Goods" covers the use of composite cylinders. However, CAN/CSA B339 does not address the new designs and materials that are becoming available in the marketplace. For example, Dynetek, a Canadian cylinder manufacturer, is using a design that includes a high strength aluminum liner and carbon reinforcing fibers, materials not currently covered under CAN/CSA B339. Therefore, it is important for Canadian input during the formation of the international standard. Powertech is now a member of the ISO Working Group 11 for composite cylinders.

Powertech has considerable experience in the formation of the ISO cylinder standard FDIS 11439, where composite cylinders are used for the storage of natural gas as an on-board fuel for vehicles. Canada and the USA would like to see the ISO requirements based on cylinders used for many years on natural gas vehicle service. Certain European interests, with their lack of experience in the use of advanced composite wrapped cylinders, want excessively conservative requirements.

The standard is divided into three parts:

ISO/CD 11119-1	Hoop wrapped composite gas cylinders
ISO/CD 11119-2	Fully wrapped fibre reinforced composite cylinders with load sharing
	metal liners
ISO/CD 11119-3	Fully wrapped fibre reinforced composite cylinders with non-metallic
	and non-load sharing metal liners

The committee drafts (CDs) were reviewed by the member countries and a large number of comments were sent back to the working group. A special meeting was held in London on March 18 and 19, 1999, to resolve the critical issues. Joe Wong of Powertech, along with Heinz Portman, president of Dynetek, represented Canada at this meeting. Twenty-one resolutions were made during this meeting to address the list of fundamental issues concerning these documents. These resolutions are described in Appendix A. The resolutions will be incorporated in a draft international standard (DIS) and circulated to member countries for voting.

3. CYLINDER INSPECTION TECHNIQUES

A dramatic increase has occurred in the number of composite-reinforced cylinder designs entering compressed gas service on board natural gas vehicles. In particular, carbon fibre designs are in greater demand because of their relatively low weight. However, certain types of glass fibres are susceptible to environmental degradation, which can result in stress corrosion cracking of the fibres. While carbon fibre designs are resistant to environmental effects, they are highly susceptible to impact damage, which lead to the eventual stress rupture failure of the fibres. While the stress corrosion cracking of glass fibres can be visually detected prior to cylinder failure, impact damage involving carbon fibres remains difficult to observe.

Efforts are under way to develop impact indicator coatings that could be applied to composite-wrapped cylinders to enhance the visual inspection of cylinders [1]. However, a qualitative method of non-destructively assessing the integrity of a composite-wrapped design is required. A non-destructive method could be used either in a periodic inspection program, for the inspection of areas of suspected damage, or in the development of an on-board diagnostic system to continually monitor the integrity of the fuel storage system.

A non-destructive inspection method would require either introducing a controlled signal into the cylinder structure, or causing the structure itself to generate a signal for analysis. Since the ideal objective would be to develop an inspection method that did not require the cylinder to be removed from the vehicle, any signal introduction method would require transmission through the entire composite structure. This is a difficult proposition because of the attenuation associated with the non-homogeneous nature (individual fibres in a resin matrix) of a composite structure.

Acousto-ultrasonics involves using a transmitting transducer to introduce a controlled ultrasonic lamb (plate) wave signal into a structure and recording the response at a receiving transducer. The change in parameters caused by interaction of the wave with the cylinder structure can be used to provide an indication of structural damage.

Previous studies had determined that AE had the potential to detect significant composite damage [2, 3]. A passive system, AE relies on the cylinder composite wrap to generate signals when it is subjected to a stress (i.e. pressurization). Acoustic emission also has the potential to differentiate between damage to the resin and the fibre structures. The amount of signal attenuation that occurs in various designs (i.e. the number of sensors required) and the criteria for acceptable or rejectable damage are unkown.

4. **RESEARCH APPROACH**

A comparison of the flaw detection abilities of acousto-ultrasonics with acoustic emission on carbon fibre wrapped cylinders was originally planned, in addition to the loan of an acousto-ultrasonic inspection unit from Digital Wave in Colorado. However, this plan could not be followed when Digital Wave decided they wanted to rent the equipment out, something that could not be accommodated by the project budget. Enquiries were made with Exponent FAA in California, regarding the availability of their acousto-ultrasonic equipment. They were of the opinion that acousto-ultrasonic signals suffer very high attenuation in composite structures, limiting their effectiveness in inspecting relatively large areas associated with cylinders. As a result of the above discussions, it was decided that the project would concentrate on using acoustic emission methods to detect damage on cylinders.

Preliminary AE tests were conducted on a Comdyne cylinder (type 3 glass fibre fully wrapped over an aluminum liner) both before and after exposure to an acid environment. The acid caused stress corrosion cracks to occur in the glass fibres. AE tests were also performed on EDO Canada cylinders (type 4 carbon fibre fully wrapped over a plastic liner) before and after the cylinders were dropped from various heights.

5. TEST METHODS

5.1 Overview

Acoustic emission studies were performed using a PAC 8000 SPARTAN AT unit manufactured by the Physical Acoustics Corporation. AE signals were collected using PAC R15 sensors connected to PAC 1220A preamplifiers. Acoustic emission data was collected by attaching a transducer to both ends of cylinders.

Testing involved hydraulically pressurizing the cylinders to the marked service pressure and holding the pressure for a period of time (e.g. 1 minute), during which AE data would be collected. This approach was intended to simulate a test method that could be practically applied using existing filling station facilities. Key features would be the need only to attach one or two transducers to a cylinder mounted in situ on a vehicle, and filling only to the service pressure.

During each AE test the cylinder was pressurized twice to its service pressure. Acoustic emission data was collected during both pressurization cycles. This approach was used to compare the emissions generated during the first pressurization cycle by the microfractures that would occur in the epoxy matrix, to the emissions generated in the second pressurization that would involve primarily the more significant fibre breakage (in the case of a damaged cylinder).

Complete details of the following tests are provided in Appendix B.

5.2 Comdyne SCC Tests

The Comdyne cylinder design has an aluminum liner fully wrapped with a glass fibre composite. Baseline AE data was collected on an undamaged cylinder that had been used in NGV service for three years. A 150 mm diameter area on the cylinder was then exposed to sulfuric acid, causing visible stress corrosion cracks to occur. After AE data was collected, the damaged cylinder was then pressure cycled 50 times to simulate repeated filling operations in service, after which AE data was again collected.

On the undamaged cylinder, the number of AE hits was low (less than 40) and the amplitude of the hits was low (less than 40 dB). During the one-minute pressure hold it was also observed that the number of new AE hits decreased significantly over time.

After acid exposure resulted in stress corrosion cracking, the damage was readily detectable by AE immediately after the damage occurred. During the initial pressurization the number of AE hits was significant even at a low pressure. During the one-minute hold some 1,888 AE hits were recorded, many with amplitudes exceeding 60 dB. This was considered indicative of fibre breakage. Finally, the number of hits continued to increase during the hold time.

After the 50 simulated filling cycles, the overall number of AE hits had decreased slightly, but a significant number of those hits still exceeded an amplitude of 40 dB. In addition, the number of hits constantly increased during the hold time at service pressure.

5.3 EDO Impact Tests

The EDO cylinder design has a plastic liner fully wrapped with a carbon fibre composite. Tests were conducted on five EDO cylinders of the 180 L (water volume) size, measuring 380 mm in diameter and 1,850 mm in length. The EDO cylinders had been used in service for approximately one year and then stored outdoors for an additional year.

Baseline AE data was collected on all five cylinders when hydraulically pressurized to the marked service pressure of 3,000 psi. Cylinders were then dropped at a 45 degree onto a concrete surface, impacting the dome ends. One cylinder dropped from a height of 2 m, one from 1.5 m, one from 1 m, and the last two from a height of 0.5 m. The cylinders were then again hydraulically pressurized while AE data was collected.

The trend of the baseline AE data was the same for all five cylinders, with the number of AE hits decreasing significantly during the second pressurization cycle. After impacting, acoustic emissions could be readily detected in all five cylinders, even at very low pressures. In all five cylinders, a high number of AE hits with high amplitudes (i.e. over 80 dB) was generated. It was found that subsequent pressure cycles would in some cases increase the AE hits, indicating that the amount of damage was being increased. Both AE sensors mounted on either end of the cylinders would detect the damage, although the indications would be more pronounced at the sensor closest to the damage.

6. **DISCUSSION**

To ensure the safe adoption of composite cylinders into the Canadian transportation market, it is important that the experience obtained from the use of composite cylinders in natural gas vehicle service be used in the development of ISO 11119 standard. At the ISO special meeting, Canadian concerns were addressed and resolutions passed that will allow the development of the standard to proceed. Transport Canada has indicated to Powertech that their intention is to adopt the standard once it is finalized.

It was found that in all cases the impact (drop) damage on the EDO cylinders was readily detectable by AE, although it was not possible to quantify the amount of damage. Four of the five EDO cylinders burst at less than the service pressure during AE testing, failing at the location of the impact damage. Additional testing would be required on cylinders that had incurred less severe impact damage, to ensure AE could be used to detect damage that could still affect the integrity of cylinder designs over time.

7. CONCLUSIONS

The resolutions adopted at the special meeting of ISO/TC 58/SC 3 reflected Canada's experience with the safe use of composite-wrapped cylinders in transportation applications.

Using only two sensors, AE was found capable of detecting the presence of damage to the composite wrap of the tested cylinders.

A simple non-destructive inspection method for composite-wrapped cylinders involving the filling of the cylinder to service pressure while AE data is collected for up to one minute is feasible.

8. **RECOMMENDATIONS**

Canadian participation in the development of the ISO 11119 standard for composite cylinder designs should be actively maintained in order to expedite completion of the standard, and to promote its adoption by Transport Canada.

To confirm the feasibility of using acoustic emission as a field inspection method, additional acoustic emission tests should be performed on cylinders containing smaller (less critical) amounts of impact damage, while the cylinders are installed on vehicles and pressurized using natural gas.

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- 2. Powertech Labs Inc., *Environmental Damage of Fiberglass Fully-Wrapped NGV Cylinders Due to Moisture and Road Salt Exposure*, Gas Research Institute Report GRI-94/0399, October 1994.
- 3. Powertech Labs Inc., *Condition Assessment of Glass Fiber Hoop-Wrapped Cylinders Used in NGV Service*, Gas Research Institute Report GRI-97-/0052, July 1997.

APPENDIX A

REPORT OF THE SPECIAL MEETING OF ISO TC 58/SC 3 -GAS CYLINDER DESIGN LONDON, UK - MARCH 1999

(not available in electronic format/ pas disponible en format électronique)

APPENDIX B

FINAL REPORT

ON

ACOUSTIC EMISSION TESTING

POWERTECH LABS INC.

ASSESSMENT OF THE USE OF ACOUSTIC EMISSION AS AN INSPECTION METHOD FOR FRP WRAPPED CYLINDERS

PROJECT: 11633-34

September 1999

C. T. Webster, Manager Gas Systems Group Materials Technologies

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TABLE OF CONTENTS

			Page
1.0		INTRODUCTION	3
2.0		WORK PERFORMED	3
	2.1	Introduction to Acoustic Emission	3
	2.2	Stress Corrosion Test	4
	2.3	Study of Impact Damaged Cylinders	17
	2.4	Discussion	68
3.0		CONCLUSION	70
	ACI	KNOWLEDGEMENT	71
	APF	PENDIX – Generalities About Acoustic Emission	

1.0 INTRODUCTION

The increasing use of natural gas as a fuel for vehicles over the past decades has lead to the use of fiber reinforced plastic in the design of natural gas vehicle (NGV) gas cylinders.

To ensure the safety of the users, it is necessary to maintain the structural integrity of the cylinders. But the actual inspection techniques, derived from the standards issued for the inspection of steel cylinders are not adapted to these new materials. It is necessary to define new inspection techniques.

The use of nondestructive testing methods such as ultrasonic scanning offer a great potential but require removal of the cylinders from the vehicles. They are therefore time-consuming, expensive, and consequently difficult to use.

Acoustic emission (AE), largely used in the industry as an inspection method for pipes and pressure vessels present the ability to be used as an on board inspection technique for NGV cylinders, offering a simple, time effective method of inspection.

The present work was done to determine the ability of the acoustic emission technique to detect damage to a composite cylinder through a simple test: monitoring the acoustic emission while filling the cylinder to its service pressure.

Two main experiments were conducted, relying on different cylinders: the study of a cylinder undergoing a stress corrosion test, and a study of five cylinders, submitted to drop tests from different heights.

2.0 WORK PERFORMED

2.1 Introduction to Acoustic Emission

The basics of acoustic emission are given in appendix. However, the following words are necessary to interpret and understand AE results.

- The acoustic emission counts: the number of time the acoustic signal crosses the threshold during a hit. Generally, the higher the number of counts, the most significant the hit.
- The hit amplitude: the maximum analog signal during a hit.
- The event: material change in the structure giving birth to the hit.
- The hit energy: the relative measure of the total energy of a hit, measured as the area under the amplitude-time curve.
- The felicity ratio. It is the numerical value of the load at which significant AE occurs on a subsequent cycle divided by the maximum load during the previous cycle. The lower the value, the weaker the structure.

The test used to assess the damages of the cylinders is based on the pressurization of the cylinder

to service pressure: 207 bar (3000 psi). The pressure was then held one minute. The AE is recorded from the start of the pressurization to the end of the pressure hold.



In the graphs obtained, we refer to Time as the pressurization and hold time during the particular test analyzed.

2.2 Stress Corrosion Test

2.2.1 Test Sample

The test was performed on a type 3 cylinder design manufactured by Comdyne. It is a 254 mm (10 inches) diameter cylinder made of an aluminum liner fully wrapped with fiberglass composite. The test sample was manufactured in January 1995 and was used on a vehicle for 3 years.

2.2.2 **AE Equipment**

The equipment used was a PAC 8000 SPARTAN AT acoustic emission recorder. The sensor used was a PAC R15 sensor connected to a PAC 1220A preamplifier. Dow Corning high viscosity vacuum grease provided acoustic coupling and the sensor was held in place in the middle of the test sample using elastic tape. The different settings of the system are summarized in Table 1.

Table 1: Test Setup				
Parameter	Value			
Threshold	30 dB			
Gain	40 dB			
Peak definition time	50 µs			
Hit definition time	1 ms			
Hit lockout time	600 μs			

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The time settings were determined thanks to a test based on the Shoe-Nielsen source (carbon pencil lead break). They were adapted experimentally from basic values for composites materials, in order to obtain one single hit for each lead break, wherever it occurred on the cylinder surface.

2.2.3 **Test Procedure**

Initially, the cylinder was scanned, recording the AE activity during hydraulic pressurization to the operating pressure of 207 bar (3000 psi), and then to the design service pressure of 248 bar (3600 psi). Water was used as the test fluid for the whole experiment. The average pressurization rate is 0.9 bar/s (13 psi/s).

The cylinder was then submitted to a stress corrosion test. A cotton patch soaked with sulfuric acid is applied on the surface of the cylinder. The acid used is a 30% solution by volume in water, simulating the acid of a car battery.

The cylinder was then loaded to 207 bar (3000 psi), (usual operating pressure), and held at this pressure for 100 hours.

During this time, significant damage to the composite was produced, as shown on Figure 1.



Figure 1: Damage to the Composite Wrapping

The cylinder was then pressurized from ambient pressure to 207 bar (3000 psi) and the acoustic emission activity recorded.

It was finally cycled from ambient pressure to 207 bar (3000 psi) for 50 times and examined again to evaluate the attenuation of the acoustic emission.

2.2.4 Test Results

A Preliminary Inspection

For each pressure level, the test was conducted twice. As a matter of fact, it is noticeable that the acoustic emission is less important the second time than the first, in accordance with the Kaiser effect. This can be explain by the fact that when put under stress, the composite structure redistribute stress over the time to accommodate the load. Locally, micro fractures of the matrix and fibers relieve the load, creating the AE hits. During the second test, the load being the same as in the first test, an important number of these fractures already exist, which means that less acoustic emissions are recorded.

The number of hits detected is small, less than 40, and more important their amplitude is very small either, (less than 40 dB). A low amplitude hit is attributed to cracks in the resin or rubbing noise in presence of delamination in the composite, whereas high amplitude is associated to the rupture of fibers. The events occur mainly at a high load (over 2500 psi). The display of the number of counts versus the amplitude of the signal provides us another useful information; the number of counts is very limited for each event, which confirm that they are not important, and that their energy level is low. Consequently, the felicity ratio is equal to 1, which means that the composite is not damaged. Finally, we can notice that during the one-minute hold of the pressure, the number of new hits is low. We can therefore conclude that the acoustics emissions recorded correspond to the normal noise of a composite structure and that the cylinder is intact. The acoustic emission recorded for 248 bar (3600 psi) confirmed these results showing no significant hits.



Graphs 1 to 7 present the results for the pressurization to 207 bar (3000 psi).

Graph 1: Number of hits versus time



Graph 2: Number of Hits versus Pressure Rise



Graph 3: Number of Hits versus Pressure Hold Time



Graph 4: Amplitude of Hits versus Pressure Rise Time



Graph 5: Amplitude of Hits versus Pressure Hold Time





Results After Stress Corrosion of the Cylinder

The test produced significant damages to the composite wrapping of the cylinder. Several major cracks were apparent, either at the location of the acid patch and on several other locations on the sidewall, as shown in Picture 2.



Figure 2: Damage to the Composite Wrapping

The scan of the cylinder with the AE provides very interesting data. The first difference is the considerable number of hits, 1888, compared to less than 40 on the intact cylinder. Even at very low pressures, a significant amount of acoustic emissions is recorded. This noise at low pressure has two main origins:

- Existing damage: matrix to matrix or matrix to fibers frictional noise,
- New damage in the composite, cracking of the matrix, ply separation, or fiber pullout.

As the pressure increases, the cylinder expands and the different composite layers move against each other, creating the noise. It clearly proves that the cylinder is damaged. Secondly, the sharp increase in the AE activity occurring after 150 seconds is representative of further damage occurring to the wrapping during the test. Graph 6 shows a range of events with high amplitude at that precise time, which can be attributed to fibers breaking during the test. Thirdly, it is important to notice that during the minute when the load remains constant at 207 bar (3000 psi), the number of hits recorded continues to increase at the same rate. It shows that damage is still occurring to the wrapping, though the load remains constant. It indicates that the wrapping might be overloaded, and might be subject after a certain time to stress rupture. It is characteristic of a damaged structure, insofar as no damage should be created at service pressure on a normal cylinder.

The felicity ratio corresponding to these acoustic emissions is low 0.47, which indicates that the composite has been heavily damaged The result of this test is that a monitoring the acoustic emission of the cylinder during a simple fill to the service pressure is enough to detect the damage to the structure, and to conclude that it would be unsafe to continue using this cylinder.



Graphs 7 to 12 give the record of acoustic emissions for a fill test to 207 bar (3000 psi) on the damaged cylinder.

Graph 7: Number of Hits versus Time



Graph 8: Number of Hits versus Pressure Rise



Graph 9: Number of Hits versus Pressure Hold Time



Graph 10: Amplitude versus Pressure Rise Time



Graph 11: Amplitude versus Pressure Hold Time

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Graph 12: Number of Counts versus Hit Amplitude

Results After 50 Cycles

Visual inspection of the test sample after 50 pressure cycles from 20 bar (300 psi) to 207 bar (3,000 psi) did do reveal new damage to the wrapping. The record of the acoustic emissions during the pressurization to service pressure gives interesting technical data. The trend is that there is an attenuation of the acoustic emissions after the cycling. The number of hits slightly dropped, and more important, the number of high amplitude hits is lower. But we can still notice that an important number of hits have an amplitude greater than 40 dB, whereas there was none on the undamaged cylinder.

There is still an important increase of the number of hits after 140-150 seconds, in which the amplitudes are ranging from 30 dB to as high as 62 dB as seen in Graph 16. It appears that it corresponds mainly to frictional noise due to delamination in the composite material. Moreover, Graph 15 shows that the number of hits is constantly increasing during the hold at 207 bar (3000 psi).

We can notice on Graph 17 that there is one hit of very high amplitude occurring during the hold time. This means that fibers are breaking at constant pressure.

Finally, the felicity ratio is 0.45, which allows us to state that the cylinder is strongly damaged.



Graph 13: Hits versus Time



Graph 14: Hits versus Pressure Rise


Graph 15: Hits versus Pressure Hold Time



Graph 16: Amplitude versus Pressure Rise Time



Graph 17: Amplitude versus Pressure Hold Time



Graph 18: Number of Counts versus Hit Amplitude

2.2.5 Discussion

The results of the test are encouraging. Indeed, it was possible to determine the quality of the test cylinder, through a simple fill test to the service pressure.

Moreover, the last part of the test shows that even after several fill cycles, it remains possible to determine that the cylinder is damaged. This suggests that for the test of a cylinder on board of a vehicle, it would be possible to examine it while filling it normally. It would not be necessary to over-pressurize it, nor to dismount the cylinder from the vehicle. After being scanned, the cylinder was, held for five minutes to 258 bar (3750 psi) for five minutes. Visual inspection revealed that the wrapping was almost completely broken on the location of the acid patch. The cylinder failed on the following fill test at nearly 200 bar (2900 psi), which shows that the damages were very important.

2.3 Study of Impact Damaged Cylinders

2.3.1 Test Samples

The tests were performed on EDO Literider 180-liter (water volume) cylinders. Those type 4 cylinders are composed of a high-density polyethylene liner fully wrapped with carbon-fiber/epoxy resin composite. The cylinders measure 1.85 meters (73 inches) long, have an external diameter of 0.381 meters (15 inches), and an approximate weight of 47.6 kg (105 pounds), with a designed service pressure of 248 bar (3600 psi). The five test samples were manufactured in 1996, and were used in service for less than 1 year.

2.3.2. **AE Equipment**

The equipment used was a PAC 8000 SPARTAN AT acoustic emission recorder. The sensors used were two PAC R6 sensors connected to PAC 1220A preamplifiers. Dow Corning high viscosity vacuum grease provided acoustic coupling and the sensors were held in place at both ends of the test sample using elastic tape. The settings for both channels were identical and are summarized in Table 3.

Table 5. Test setup		
Parameter	Value	
Threshold	50 dB	
Gain	30 dB	
Peak definition time	50 μs	
Hit definition time	400 μs	
Hit lockout time	600 μs	

 Table 3 : Test setup

Detail of the location of both sensors is given in Figure 3.



Figure 3: Location of the sensors

The first sensor is mounted on the bottom end of the cylinder, near the first impact area, and the second one on the valve end, near the second impact area, due to the rebound.

2.3.3. Test Procedure

Prior to the experiments, each cylinder was filled to 207 bar (3000 psi) three times and the acoustic emissions recorded, to detect any pre-existing default or abnormality. The pressurization rate for all the tests is 0.2 bar/s (3 psi/s). Each cylinder was then submitted to a drop test. The test samples were dropped from a different height at a 45 degree angle. The height is expressed from the lower end of the cylinder to the ground. The test samples were free to rebound. The test parameters are summarized in Table 2.

Experimental parameters			
Serial number	Weight (kg)	Drop height (m)	Impact energy (J)
9622004	49.9	0.5	565.7
9620219	49.9	0.5	565.7
9622010	47	1	763.3
9611220	52.5	1.5	1110.2
9611269	49.5	2	1289.5

Table 2 : Drop Test Parameters

Finally, each cylinder was filled to 207 bar (3000 psi) and the acoustic emissions recorded, to determine the influence of the drop on the cylinder AE response. This will help to assess the liability of the acoustic emission testing of cylinder during pressurization to service pressure.

2.3.4. Test Results

For each cylinder, the drop provoked important damage to the wrapping, as shown by Figure 4, with apparent cracks and delamination.



Figure 4: Damage Due to Cylinder Drop.

Visual inspection revealed that for each cylinder, the damaged area is more important for the second hit, after rebound. This can be seen by the size of the damaged surface as well as the length of the cracks in the superficial layers of the wrapping.

a) 2m Drop

Preliminary Inspection

The inspection shows that the cylinder is undamaged. The acoustic emission is low until 193 bar (2800 psi), the response of both sensors is almost identical, and the felicity ratio is good: 0,91. Most of the activity occurs at the higher pressure. We can also notice that the range of the amplitude is quite broad. This can be explained by the fact that carbon fibers generally give greater amplitude to the hits than glass fibers. Moreover, there is no autofrettage on type 4 cylinders. This means that the liner can move inside the wrapping, creating rubbing noise.



Graph 19: Hits versus Time for Both Sensors



Graph 20: Hits versus Time for Sensor 1



Graph 21: Hits versus Time for Sensor 2



Graph 22: Hits versus Pressure Rise



Graph 23: Hits versus Pressure Hold Time



Graph 24: Amplitude versus Pressure Rise Time



Graph 25: Amplitude versus Pressure Hold Time

Post Drop Inspection

The cylinder failed during the first pressurization at 156 bar (2270 psi). However, the inspection of the acoustic emissions reveals some interesting points. The examination of the graphs reveals that there is a burst of AE at the very beginning of the test. For pressures still as low as 7 bar (100 psi), there is already about 2000 hits. These hits can be associated with the rubbing noise due to the delaminations inside the wrapping. With the increasing pressure, the cylinder expands, provoking this burst of events. It is characteristic of a damaged structure. It is obvious that the cylinder is damaged. The felicity ratio dropped to 0.02, which means that the material has lost almost all its strength.

The analysis also reveals that the number of hits is more important for the second sensor. This corresponds to the importance of the damages, and finally, the failure occurred on the spot of the second hit. There is a correspondence between the importance of the damage and the number of hits. The analysis of the amplitudes confirms the previous observations. We can notice an important number of hits of relatively low amplitude at the beginning of the pressurization. It corresponds to the frictional noise mentioned above. Then, the higher the pressure, the higher the amplitude of the hits, which shows that heavy damage is occurring to the composite during the test. It is possible to detect that the cylinder is damaged.



Graph 26 : Hits versus Time for Both Sensors



Graph 27: Hits versus Time for Sensor 1



Graph 28: Hits versus Time for Sensor 2







Graph 30: Amplitude of Hits versus Time

b) 1.5 m drop

Preliminary inspection

The inspection of the cylinder shows that it is undamaged. Indeed, the total amount of signals is low (about 100), the activity occurs at the highest pressure and the felicity ratio is good: 0.97. Moreover, we can notice that the activity slows down during the hold time.



Graph 31: Hits versus Time for Both Sensors



Graph 32: Hits versus Time for Sensor 1



Graph 33: Hits versus Time for Sensor 2



Graph 34: Hits versus Pressure Rise



Graph 35: Hits versus Pressure Hold Time

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Graph 36: Amplitude Hits versus Pressure Rise Time



Graph 37: Amplitude versus Pressure Hold Time

Post Drop Inspection

The cylinder failed during the first pressurization after the drop test. The pressure at the failure was 156 bar (2260 psi). The examination of the curves reveals the same phenomenon as for the previous sample. A burst of acoustic emissions occurs at the very beginning of the test, but for sensor 1 only. It is less important but still shows that there is damage to the wrapping. The plot of the amplitudes confirms this showing an important number of hits at the very beginning of the test, and then a quieter period, before the increase of either the number of hits and their amplitude. The felicity ratio is 0.14, which confirms the poor quality of the material. It is interesting to notice that the number of hits is more important for the sensor 2 than for sensor 1, which is in accordance with the importance of the damage. The study of the amplitudes are higher than 80 dB and 90 dB. This is characteristic of fibers breaking, even at these low pressures. The results are displayed in the following set of graphs.



Graph 38: Hits versus Time for Both Sensors



Graph 39: Hits versus Time for Sensor 1



Graph 40: Hits versus Time for Sensor 2



Graph 41: Hits versus Load



Graph 42: Amplitude of Hits versus Time

c) 1 m Drop

Preliminary Inspection

The results are typical for a good cylinder. The number of hits is a bit high, but they only occur after 190 bar (2750 psi). The felicity ratio is good : 0.92.



Graph 43: Hits versus Time for Both Sensors



Graph 44: Hits versus Time for Sensor 1



Graph 45: Hits versus Time for Sensor 2



Graph 46: Hits versus Pressure Rise



Graph 47: Hits versus Pressure Hold Time



Graph 48: Amplitude versus Pressure Rise Time

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Graph 49: Amplitude versus Pressure Hold Time

Post Drop Inspection

The cylinder also failed during the first pressurization. The burst pressure was 129 bar (1870 psi). The failure occurred on the spot of the second hit, as for the two previous cylinders.



Figure 5: Failure of the Cylinder

The global number of hits recorded is considerably smaller than with the previous test

samples. But we can still notice that at the beginning of the test, the number of hits increases slightly faster, due to friction noise created by the delaminations inside the wrapping. Hits of high amplitude (100 dB) are recorded very early in the test, and the felicity ratio is still very low at 0.15, which clearly shows that the structure is damaged. The observation of the amplitudes shows that a high number of event with a very high amplitude start to appear very soon in the test, showing that the structure has been damaged.



Graph 50: Hits versus Time for Both Sensors



Graph 51: Hits versus Time for Sensor 1



Graph 52: Hits versus Time for Sensor 2



Graph 53: Hits versus Load





d) 0.5 m drop : Cylinder 9620219

Preliminary Inspection

The test shows that the acoustic emissions recorded correspond to an undamaged cylinder. Activity starts over 180 bar (1800 psi), and the felicity ratio is good: 0.9. The amplitudes recorded remain under 90 dB, which shows that there are no major events.



Graph 55: Hits versus Time for Both Sensors



Graph 56: Hits versus Time for Sensor 1



Graph 57: Hits versus Time for Sensor 2



Graph 58: Hits versus Pressure Rise



Graph 59: Hits versus Pressure Hold Time



Graph 60: Amplitude versus Pressure Rise Time



Graph 61: Amplitude versus Pressure Hold Time

Post Drop Inspection

• First Inspection

We can see that the number of hits recorded by the second sensor is considerably bigger than the one of the first sensor. This would correspond with the importance of the damages to the structure. The test results clearly show that the cylinder is damaged. The first hits appear at very low pressure, and their amplitude become very high (84 dB) at pressures as low as 68 bar (990 psi). The global number of hits is very important in comparison with a non-damaged cylinder, and the felicity ratio dropped to 0.33.



Graph 62: Hits versus Time for Both Sensors



Graph 63: Hits versus Time for Sensor 1



Graph 64: Hits versus Time for Sensor 2



Graph 65: Hits versus Pressure Rise



Graph 66: Hits versus Pressure Hold Time



Graph 67: Amplitude versus Pressure Rise Time



Graph 68: Amplitude versus Pressure Hold Time

• Second inspection

The results show that the general number of hits diminished a lot. Nevertheless, the trend remains characteristic of a damaged cylinder, with a quick rise at the beginning associated with delaminations in the wrapping. The felicity ratio is 0.42, which is still low and indicates the poor mechanical properties of the material.

The examination of the amplitudes reveals that there are fewer event of high amplitude than for the first pressurization. But, several hits with an amplitude higher than 90 dB confirm that the structure is damaged.



Graph 69: Hits versus Time for Both Sensors



Graph 70: Hits versus Time for Sensor 1

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Graph 71: Hits versus Time for Sensor 2



Graph 72: Hits versus Pressure Rise



Graph 73: Hits versus Pressure Rise Time



Graph 74: Amplitude versus Pressure Rise Time



Graph 75: Amplitudes versus Pressure Hold Time

• Third Inspection

We can still easily detect the damages to the cylinder. The fast increase in the number of hits at low pressure and the number of hits are characteristic. The value of the felicity ratio determined is 0.2, which is still very low. The graph of the amplitudes confirms the previous statement, showing the important rubbing noise at the beginning of the pressurization, and few high amplitude events at high pressure.



Graph 76: Hits versus Time for Both Sensors


Graph 77: Hits versus Time for Sensor 1



Graph 78: Hits versus Time for Sensor 2



Graph 79: Hits versus Pressure Rise



Graph 80: Hits versus Pressure Hold Time



Graph 81: Amplitudes versus Pressure Rise Time



Graph 82: Amplitudes versus Pressure Hold Time

It is interesting to notice that there is a big attenuation of the AE signal between the first pressurization and the other ones. But however strong it is, we are still able to detect the damage to the structure thanks to the shape of the plot, as well as the felicity ratio. Another interesting fact is that the damages are more obvious in the last test than in the second. This means that some further damage occurs during each test, and that the cylinder is more and more likely to fail under stress.

Finally, the cylinder was submitted to a burst test and failed at 328 bar (4757 psi). The failure occurred on the spot of the second impact. The low burst pressure testifies that the cylinder was heavily damaged.

e) 0.5 m drop: cylinder 9622004

Preliminary Inspection

The test results are normal. We have almost no events under 193 bar (2800 psi), and the felicity ratio is 0.89, which is still good. Though we notice some events of high amplitude, the cylinder appears to be undamaged.



Graph 83: Hits versus Time for Both Sensors



Graph 84: Hits versus Time for Sensor 1



Graph 85: Hits versus Time for Sensor 2



Graph 8: Hits versus Pressure Rise



Graph 87: Hits versus Pressure Hold Time



Graph 88: Amplitude versus Pressure Rise Time



Graph 89: Amplitude versus Pressure Hold Time

Post drop Inspection

• First Inspection

The results are quite characteristic of a damaged cylinder, with rubbing noise at the beginning, a high number of events, and no slow down during the minute at constant pressure. The acoustic emissions start at very low pressure, as can be seen on graph 94. The felicity ratio is 0.05. We can notice a very high number of hits which amplitude is over 90 dB.



Graph 90: Hits versus Time for Both Sensors



Graph 91: Hits versus Time for Sensor 1



Graph 92: Hits versus Time for Sensor 2



Graph 93: Hits versus Pressure Rise



Graph 94: Hits versus Pressure Hold



Graph 95 : Amplitude versus Pressure Rise Time



Graph 96: Amplitude versus Pressure Hold Time

• Second Inspection

The results of the second test correspond to what could be expected, with fewer hits, but still characteristic of a damaged cylinder.



Graph 97: Hits versus Time for Both Sensors



Graph 98: Hits versus Time for Sensor 1



Graph 99: Hits versus Time for Sensor 2



Graph 100: Hits versus Pressure Rise



Graph 101: Hits versus Pressure Hold Time



Graph 102: Amplitude versus Pressure Rise Time



Graph 103: Amplitude versus Pressure Hold Time

Finally, the results for the third pressurization are quite similar, as shown by the next set of graphs. They show an important friction noise at the beginning of the pressurization, and hardly any attenuation at constant pressure. The cylinder is damaged. The felicity ratio is 0.07, which is very low. The plot of amplitude corresponds exactly to that analysis, displaying an important number of hits at the beginning and then, several hits of very high amplitude at the highest pressure.



Graph 104: Hits versus Time for Both Sensors



Graph 105: Hits versus Time for Sensor 1



Graph 106: Hits versus Time for Sensor 2



Graph 107: Hits versus Pressure Rise



Graph 108: Hits versus Pressure Hold Time



Graph 109: Amplitude versus Pressure Rise Time



Graph 110: Amplitude versus Pressure Hold Time

The attenuation between the first and the third test is important. The number of hits went from 25948 to 4326. Nevertheless, it is still obvious in the third test that the cylinder is damaged and should not be used in service anymore. The felicity ratio and the shape of the curves are clearly sufficient, and proved to be reliable indicators of the damage.

Generally, the number of hits for the second sensor is more important than for the first sensor for each inspection. It is therefore possible to say that the damages are more important on the side of the second sensor. This was confirmed when the cylinder was submitted to a burst test. It failed at 251 bar (3643 psi), on the precise spot of the second impact.

2.4 Discussion

2.4.1 Parameters Setting

The time parameters for the acoustic emission testing depend typically on the material tested. For composites, standard values given in the literature are 100 to 200 μ s for the hit definition time, 20 to 50 μ s for the peak definition time and 300 μ s for the hit lockout time. These values have to be adapted to the test material. For the present tests, this was done using a pencil carbon lead break test. The equipment used corresponds to the specifications of the ASTM E-976 standard. The settings were adapted to obtain one precise hit for each lead break at different distances from the sensor.

The major problem of this technique is that it requires a long time and numerous tests.

Consequently, to use acoustic emission in a general test method for gas cylinder, it would be necessary to develop a faster technique to adjust the time settings.

2.4.2. <u>Attenuation of the Signal</u>

For safety consideration and convenience, the tests were performed with water. In order to evaluate the influence of the test fluid on the attenuation of the acoustic emissions, an attenuation test was performed to record the attenuation of the signal on a cylinder filled with water and with air, at ambient pressure. Two EDO cylinders were examined. For each test fluid, noise was created using the lead break technique, at an increasing distance from the sensor, and the amplitude of the hits was recorded. This gave the average attenuation of the signal with the distance, depending on the test fluid. The values are summarized in Table 5.

		8
	Test fluid	Attenuation (dB/m)
Cylinder 1	Water	17.5
	Air	14.2
Cylinder 2	Water	18
	Air	10.7

 Table 5 : Attenuation Factor Depending on the Test Fluid

The results show that the attenuation is less important using air than water. It should be so with natural gas instead of air. This can be explained by the fact that water conducts the acoustic waves, creating a diffraction of the signal at the contact surface with the cylinder wall. This consequently weakens the signal. Contrarily, gases are poor conductors for the acoustic waves, which travel mainly inside the composite wall, there is less attenuation. It is logical to state that the results obtained with water could be obtained using natural gas, even with a better precision. But it will be necessary to realize further tests using natural gas to confirm it.

Moreover, the test was only performed at ambient pressure, and it will also be necessary to realize tests at the service pressure, to ensure that no major changes occur.

2.4.3. Comparison of the 0.5 m Results

It is difficult to draw a real comparison between the two cylinders 9620219 and 9622004. Though the energy of impact was the same, the number of hits recorded are different and the burst pressure also. It is consequently not possible to determine a possible correlation between the number of hits and the amount of damage. There appears to be one because the noise is more important for the second sensor for each cylinder, but it cannot precisely be established.

Firstly, the differences between the test results for each cylinder can be explained by the different quality of the wrapping for each cylinder. There was indeed a high level of

porosity for each cylinder, which indicates that the quality of the wrapping was not constant. It also explains why the cylinder 9622010, dropped from 1 m burst at a lower pressure than the cylinders dropped from 1.5 and 2 meters.

Secondly, due to the porosity, the cylinder revealed to be very sensitive to moisture. The attenuation of the acoustic waves grows stronger with the moisture in the composite. As the tests were performed outdoors, even though in a dry bunker, differences were noticed in the attenuation, and obliged sometimes to low*e*r the threshold of the recording device. The results were later filtered to the test threshold of 55 dB. Consequently, differences were probably introduced, which can explain the results.

3.0 CONCLUSION

The test performed provided very significant information. Indeed, however damaged the test samples were, it was always possible to detect the damages with the acoustic emission during a pressurization to service pressure with a one minute hold at the service pressure. It should consequently be possible to settle a simple test using acoustic emission to inspect cylinders on board of the vehicle while refueling at the gas stations.

In order to precise these results and to develop the test method, further investigation will be necessary. It is necessary to study the behavior of the cylinders filled with natural gas, to test them in real condition. The influence of the mounting brackets should be determined, as they will probably produce supplementary background noise.

Finally, the test should be performed on cylinders of consistent quality, and more important, with less important damages. This would make it possible to determine the ability of acoustic emission to detect small damaged to the composite structure, and to compare the results for the different test specimens.

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APPENDIX

Generalities About Acoustic Emission

<u>Theory</u>

Acoustic emission is a nondestructive technique for materials evaluation. It relies on the detection of elastic waves generated by sudden deformations in stressed materials. The waves travel from the source to the sensor(s), where they are converted to electrical signals. The AE instrumentation measures these signals and produces data displays from which the condition and behavior of the structure can be evaluated.

With acoustic emission, growing defects are the source of the signals, which can travel long distances to the detecting sensors. The defect can be detected remotely and moreover, the source can be located by computing the different arrival times of the wave at different sensors.



Figure A1: Principle of Acoustic Emission Testing

Settings

The settings are the key to a good test. They define the way the system will process the signals and interpret them. The first important parameter is the sensitivity. It refers to the ability of the system to detect small signals in the structure. In part, this is determined by sensor spacing and sensitivity, and in part, by the controls set on the main equipment. The main control is the Threshold, the operator-set voltage level against which the amplified AE signals are compared. When the signal exceeds this voltage threshold, a hit is recognized and the signal measurement circuitry comes into play. The threshold is specified in decibels (dB) relative to a 1-microvolt signal at the sensor.

The higher the threshold, the lower the sensitivity, as indicated in the following table.

Table 0 : System Sensitivity		
Threshold	Sensitivity	
Below 25 dB	Very high	
25-35 dB	High	
35-45 dB	Medium	
45-55 dB	Low	
Above 55 dB	Very low	

Table 6 : System Sensitivity

The system timing parameters are the second key to a good test. The signal coming from the

sensor is amplified, filtered and then presented to the measurement circuitry. The measurement process begins when the signal first crosses the threshold. It triggers assorted timers depending on the operation of the particular AE system. When the signal has passed, the circuitry must close out its operation, output the resulting hit description and get ready for the next hit.

So, the first non-trivial task is to determine when the signal has passed. It is done thanks to a timer and the Hit Definition Time (HDT). It is turned on by the first crossing of the threshold and retriggered by each new threshold crossing. It turns off when the Hit Definition Time is has expired without anymore threshold crossing.

The second task is to make sure a hit is recorded only one time. Indeed, the wave created by one event travels in every directions in the material and creates echoes that reach the sensor shortly after the first hit. This is done with a second timer and the Hit Lockout Time (HLT). The timer is triggered when the hit definition time expires, and the system discards all the incoming signals until the hit lockout time expires. That way, the echoes are not recorded as separate hits.

Finally, a third timer is used, using the Peak Definition Time (PDT). It is used to determine the rise time of a hit. It is triggered by the first crossing of the threshold and corresponds to the period we allow the system to scan to determine the signal peak.

Important Parameters

The main parameters of an acoustic emission signal are:

- The acoustic emission counts: the number of time the acoustic signal crosses the threshold during a hit. Generally, the higher the number of counts, the most significant the hit.
- The hit amplitude: the maximum analog signal during a hit.
- The event: material change in the structure giving birth to the hit.
- The hit energy: the relative measure of the total energy of a hit, measured as the area under the amplitude-time curve.

Important Factors

The following factors are to be taken into account when analyzing the significance of the acoustic emissions recorded during a test.

- The stress level at which significant AE activity occurs. The lower the stress, the weaker the structure.
- The amplitude of the hit (and the energy). The higher the amplitude, the larger the event. In a composite, high amplitude is associated with fiber rupture.
- The total number of hits. The larger the number of hits, the larger the damage to the composite.
- The felicity ratio. It is the numerical value of the load at which significant AE occurs on a subsequent cycle divided by the maximum load during the previous cycle. The lower the value, the weaker the structure.

Finally, permanent observation is necessary during a test to notice anything unusual that could influence the result of the test.