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ACOUSTIC EMISSION ANALYSIS--A TEST METHOD FOR METAL JOINTS BONDED BY ADHESIVES

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16. Abstract An investigation was conducted concerning the suitability of the acoustic emission analysis for a study of adhesive joints, which had been subjected to mechanical and climatic stresses, taking into account conditions which would make the results applicable to adhesive joints used in aerospace technology. Specimens consisting of the alloy AlMgSi-0.5 were used in the investigation together with a phenolic resin adhesive, an epoxy resin modified with a polyamide, and an epoxy resin modified with a nitrile. On the basis of the obtained results it is concluded that the acoustic emission analysis provides valuable information concerning the behavior of the considered adhesive joints under load and climatic stresses.
Among the nondestructive test methods for adhesive joints are primarily ultrasonic methods, for instance the pulse-echo system and, much more frequently, the resonant-frequency system in form of the so-called FOKKER-BOND-TESTER. In addition, some adhesive-bonded systems can also be tested through heat flow methods, holography and x-rays (1). However, fault detection is restricted to a range of few millimeters so that initial microscopic damage development in mechanically or climatically stressed adhesive bonds, which is visually already recognizable through the fracture planes and has decisive importance for the overall performance of the bond, is not recognizable or only barely so.

These test methods also do not permit conclusions as to the actual strength characteristics of an adhesive joint, which can exhibit considerable variations even in the absence of bubbles and pores in the layers of the adhesives and in a complete bond between adhesive and bonded material. This is due primarily to the structuring effect of the metal surfaces in adhesive areas close to the boundary layers (2). Finally, the application of these test methods often presents difficulties since large bonded areas in particular cannot be tested integrally but must be checked point by point in a grid-like fashion.

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1. Introduction

The method of acoustic emission analysis (SEA), which has lately been tested on metals and composite materials is not subject to these restrictions (3 to 5). On the one hand, we are dealing here with an integral test method where a few sensors permit easy checking of larger pieces like aircraft compartments or other containers and where the onset of faults can be recognized immediately. Beyond this the acoustic emission analysis can differentiate between inherent material faults, which do not spread and are therefore harmless, and fractures which can be very small on occasion but do spread, since only such faults cause acoustic emission. During measurement of the acoustic emission the component must be subjected to mechanical stress so that the method cannot be called a completely nondestructive test in the strictest sense. But these stresses can be way below the normal service stresses in several cases so that critical stresses, with potential destruction of the test body or component, are not required in the application of acoustic emission measurement.

It seemed promising to use this measurement technique for the investigation of damage cycles in adhesive bonds, caused by mechanical or climatic stresses, and to ascertain in what way ageing processes in adhesive bonds of metals generate a change in behavior of the material under mechanical stress.

2. Experimental Materials, Sample Types

The experimental materials were chosen so that the results would be directly applicable to adhesive bonding for aircraft, since the highest requirements of strength and stability for adhesive bonds of metals are presently made by the aeronautical
technology. Only an aluminum alloy could, therefore, be considered as material for bonding and AlMgSi-0.5 was shown to be most suitable for the test samples.

Three adhesives now in use with the aircraft industry were chosen because of the variations in their strength performance, deformation ability and resistance to ageing. They are:
-- the phenolic resin adhesive REDUX 775,
-- the epoxy resin FM 1000 modified with POLYAMID 66,
-- the epoxy resin AF 126 modified with nitrile.

In contrast to the REDUX adhesive, which has been used in aircraft construction for about 20 years because of its high resistance to ageing, epoxy resin FM 1000 exhibits a much higher capability for plastic deformation (6). But its resistance to ageing is not sufficient for all requirements. Compared with the adhesives mentioned, the epoxy-nitrile adhesive AF 126 is characterized by lower curing temperature, as well as better plastic deformation than REDUX 775 and with it higher resistance to peeling. When compared to adhesive FM 1000, AF 126 exhibits lower creep tendencies and higher strength at elevated temperatures and in muggy climates.

The surface preparation of the bonding components consisted of:
-- the pickling process, 30 minutes of pickling the surfaces of the bonding components in chromosulfuric acid at 60°C,
-- the pickling process with subsequent anodizing in chromic acid at direct current increasing to 40 V.

The subsequent anodizing in chromic acid has been proven, in contrast to the pickling process, to provide improved stability of adhesion in boundary layers when subject to the influx of humidity.
In the choice of sample types it had to be considered that microscopic and macroscopic fissuring processes, originating from a simulated fault, were to be generated over an extended time interval in a layer of adhesive; they were to be sensed mechanically, resp. electrically and by means of acoustic emission analysis and their growth, resp. progress, was to be observed through both measurement techniques, in a comparative way, under normal as well as aged conditions. Preliminary experiments showed that the simple lap-joint of the tensile-shear stress test was not suitable. It seemed more appropriate to choose a test sample for fissure opening, borrowed from fracture mechanics and described elsewhere (7), which consisted in this case of aluminum rods of 250 mm length, 30 mm width and 15 mm thickness. Two each were to be bonded along the 250 x 30 mm surface to a length of 200 mm, after prior appropriate surface preparation, with the adhesive to be investigated. The test load was directed at the bonding components at right angle to the bonded surface in the unbonded area, with a 0.2 mm/min. feed of the test equipment. During the bending apart of the bonding components, which is carried out similar to a peeling test, the fissuring progress begins, starting from a foil of polytetrafluoroethylene (Teflon) contained in the adhesive layer as simulated flaw. The progress of the fissure in the adhesive can be recorded by suitable odometric instruments, based on the flaring out of the bonded components at the interface of the samples, which is the direction in which the fissure in the adhesive proceeds.

3. Carrying Out the Experiment

Fig. 1 discloses the nature of the test body and its placement in the test apparatus. In this experiment the force-flaring-diagram will be measured. After a certain fissure
length has been reached the opening path of the fissure can be fixed, i.e. the fissure conserved in its flared out condition. In this prestressed condition it is possible to subject the samples to an ageing process in a muggy climate, leading in most cases to an automatic increase in the fissure length. This fissure growth usually terminates, however, after 20 to 30 hours due to the progressive decrease of load at the leading edge of the fissure, based on reduction in the flaring of the bonded components with a constant prestress path and increasing fissure length. In the prestressed condition ageing processes in the adhesive occur after rather short time intervals, as is known from the literature (8), so that an ageing time of 40 hours in muggy climate is already sufficient for the observation of marked changes in the adhesive during subsequent continuation of the fissure (7).

Acoustic emission sensors were placed on the samples in various locations during the experiments, as recognizable in Fig. 1, and the emission characteristics of normal and aged layers of adhesive were measured for the entire period of the test. A significant problem is presented here by the disturbing noise signals caused by the test apparatus, which could not be totally eliminated. During investigations of fracture mechanics they are caused primarily by friction in the bearings of the clamping fixture and by noises within the tensile stress machine. The choice of suitable input thresholds on the one hand and the simultaneous measurement of machine noises during the tensile test can, however, eliminate them. In addition, the generation of noises in clamping fixtures can be reduced greatly through the use of appropriate lubricants. On the other hand, the attempt to suppress the disturbing noises caused by the environmental chamber, during the climatic ageing of the clamped down samples, to the extent that the acoustic pulses
generated during the progress of the fissure under prestress would become available, was unsuccessful. This remains to be done in a future experiment.

Several piezoelectric quartz sensors were used for the recording of acoustic signals emitted under load in adhesive bonds. Preliminary tests showed that they must be coupled to the contact surface via some incompressible fat. Disturbing signals from machines and from the environment could be eliminated by only measuring signals in the ultrasonic frequency band. In the present case that was between 100 kHz and 2 MHz (broadband) for frequency analyses and around 140 kHz for the formation of acoustic sums and analyses of the pulse area. Resonant sensors proved best for the latter procedures because of their higher sensitivity in measurements.

Fig. 1. Test body (above) and test arrangement (below) for the acoustic emission analysis.
4. Experimental Results

The acoustic sum determined each time could be recorded jointly with the curve for opening under force, in Fig. 2. It must be stressed here that further filtering out of possible interference noise signals permitted an additional operative discriminator threshold which released only signals above 100 mV amplifier input voltage for recording. During the tests the pulse cam distribution was recorded by a multi-channel counter, with individually recorded pulses assigned to different channels according to their respective cams. To carry out the frequency analysis it was necessary to store the signals and then feed them into a frequency analyzer. Comparative preliminary experiments with tensile-shear test samples, as well as with the described samples from fracture mechanics, allowed a safe deduction that the acoustic signals so obtained and recorded are clearly to be traced to the fissure formation in the adhesive, resp. between adhesive layer and bonding material, while the other, suppressed, noises were either disturbing or unimportant in the interpretation of the results.

4.1 Fissuring Process and Acoustic Sum

Looking at first at the curves of opening under force, which were obtained from unaged and aged adhesive joints, it must be stated that in the unaged condition a clear maximum is reached after a nearly linear increase in force where the fissuring progress apparently proceeds macroscopically from the Teflon foil embedded in the adhesive layer, involving naturally a decrease in force with constant opening velocity. The maximum force required for fissuring depends, as in the peeling test, on the type of adhesive, i.e. primarily on its deformation capability. As compared to the relatively brittle adhesive Redux both the highly plasticized FM 1000, as well as the
Fig. 2. Sums of pulses $\Sigma J$ and curves of openings under force
Adhesive: a,b: REDUX 775; c: FM 1000; d: AF 126
Pretreatment of surface: a: "pickling"; b to d: "pickling" and anodizing
Amplification: a,b: 70 db; c,d: 80 db.
epoxy resin, require increased force for fissuring. It is remarkable in this connection that for the adhesive REDUX the fissuring force is apparently also significantly influenced by the preliminary surface treatment of the metallic bonding components. It must be emphasized that in both cases the fissure proceeded as cohesion fracture in the adhesive layer. The type of preliminary surface treatment apparently also has influence in these test bodies not only on the characteristics of the boundary layer area but beyond it on those of the entire adhesive layer.

After a certain opening length was reached the samples were, as mentioned, placed in clamped condition in a muggy climate of 40/95 (40°C, 95% relative humidity) for a time interval of 40 hours. Subsequent experiments continuing the fissure were carried out with the results also recorded in the diagrams. During continuation of the fissuring a definite force maximum, similar to the one in the unaged condition, can still be observed in several cases but the increase in force is not as steep, which may be laid to adhesion fractures that occur only partially at first. In the evaluation of the test results it should not be overlooked that during the ageing tests of the clamped fissure it often moves from the adhesive layer into the boundary layer. After the climatic exposure and the fissuring progress in its course, in the form of an adhesion fracture, there is apparently still a zone of weakened adhesion in the unfissured area which must first be overcome during an experiment of continued fissuring. If the fracture continues again in the adhesive, during continuation of the fissure, then it must not be overlooked that the decrease in force after attainment of the maximum, particularly for the adhesives REDUX and FM 1000, is less steep in the aged condition than when unaged. This permits the conclusion that the adhesive layer is
plastified by infused humidity and so contributes to stabilization against further fissuring.

The curves of acoustic sums, which are placed next to the appropriate curves of opening under force, now permit additional interpretation of the failure mechanisms. In the unaged condition it must not be overlooked that acoustic signals are already emitted during the nearly linear force increase, i.e. that damages occur in the adhesive. But they do not yet cause any macroscopic fissure, which can be recognized only by a marked increase in the slope of the acoustic sum curves after the maximum of force has been reached. Only acoustic emission analysis permits a clear statement that in adhesive joints, similar to those in metallic materials, the first force maximum in experiments of fracture mechanics coincides with the beginning of a macro-fissure. This maximum force, which is required for fissure progress must not, however, be included in calculations in the form of a fracture-mechanical characteristic, since damages to the adhesive layers, though different for each adhesive, apparently occur before the initiation of the fissure.

The characteristic of acoustic emission for aged samples (always to the right in the diagrams) is different. After the force maximum has been reached the acoustic sum does quite clearly increase here, too. It permits the conclusion that the macroscopic fissure continues in the aged adhesive layer. The number of damages that occur, visible from the rise of the curves of the acoustic sums in the aged condition, varies considerably with the adhesives. This is particularly apparent when concentrating consideration on the behavior of adhesive FM 1000 which is marked by high plasticity. This adhesive, which apparently has been further plastified by humidity, shows only an indistinct fissure start in the aged condition, which is made clear by the curves of opening under force and also by the very
slow rise in the acoustic emissions curve. Even for relatively high loads does this adhesive, therefore, provide great assurance against fissure continuation even in the aged condition. The increasing opening of the joint components is met by this adhesive through plastic deformation at constant resistance which, as noticeable in the curve of the acoustic sums, is marked by only a few irreversible damage phenomena. Adhesive FM 1000 which, according to other tests (6), shows relatively little resistance to ageing compared to AF 126, is also distinguished by its improvement in resistance to the progress of a fissure under the influence of humidity in the case of some damage in the adhesive layer. Even for relatively extensive plastic deformations, such as occur in buckling sheet-metal constructions for instance, the deformation will hardly cause irreversible damages that could contribute to further unstable fissuring.

Acoustic emissions which could be recorded from the unaged adhesive layers, to the left in the diagrams, already before the generation of the macroscopic crack and which permit conclusions about the damages in the adhesive layer, do not appear in the aged condition. The summation curves rise here markedly only after the force maximum has been reached. Two explanations can serve as interpretations for the shape of these curves: on the one hand it is possible that the boundary layer, which has apparently been weakened by humidity in the clamped condition, fails under emission of only a few and weak acoustic signals until the fracture moves over again clearly into the adhesive layer. On the other hand it is conceivable that the adhesive, which has been more plastified during the 40 hour climatic treatment, particularly in the vicinity of the leading edge of the fixture, follows at first the opening in the joint components and that irreversible damage cycles do not occur during this plastic deformation. An analogy to this would be found
in the behavior of the adhesive FM 1000 with only a few damage cycles in the aged condition. The fact of absent emission up to the macroscopical crack in the aged condition permits the conclusion that all adhesives gain increased resistance to irreversible destructive processes through the infusion of water (made clear in most cases through a more gradual rise in the slope of the acoustic sum curves). Only a small number of damage mechanisms seem to occur apparently in the adhesive layers during continued fissuring. Plastic deformations, which appear to be supported by the influence of humidity on adhesives, do create an increase in safety then, to some extent.

To provide some insight into the reproducibility of the experiments described characteristics for adhesive AF 126, as determined for two different samples, have been plotted in Fig. 2d. These are the greatest differences found for the adhesive. The acoustic summation curves are nearly identical while those for opening under force differ considerably in the unaged condition. If we stay with the hypothesis, for the time being, that plastic adhesive deformations are not connected with damage cycles in the microscopic or submicroscopic area and consequently do not cause acoustic emissions, the difference in behavior of the force path for an identical emission picture can be explained by a higher degree of plasticity in sample 1 being responsible for the higher resistance to fissuring, which can safely be accepted (1). The higher degree of plasticity in sample 1 could be attributed to the inhomogeneity of the adhesive used, which is not completely avoidable in the production of adhesives. The higher plasticity of the adhesive tested in sample 1, i.e. a higher percentage of thermoplastic resin that is added for plastification, would then also be responsible in the aged condition for the slope of the acoustic summation curve rising less steeply than that of sample 2 with a smaller share of plastifying resin, after continuation of the
fissure has started. The interpretation presented here cannot be generalized so far, however, since there are not yet sufficient experimental data available to support it. But one can already recognize from this example that the ratio in which the different adhesive components are mixed, namely the cross-linking duroplastic ones and the plastifying, not cross-linked, thermoplastic ones, is of decisive importance in the behavior of the fissure.

The fissuring characteristic of an adhesive varies clearly with the application of different methods of surface preparation, as shown in Fig. 2 a,b for the REDUX adhesive. This is confirmed by the acoustic emission characteristic which points to a larger number of damage cycles in the adhesive after pickling and anodizing than after pickling alone, which is also combined with a markedly higher overall load-carrying capacity at the same time. This could be attributed to the fact that the remote effect of the metal surfaces in the case of pickling and anodizing provides a higher degree of orientation, at least for the duroplastic component of the phenolic resin, or a higher degree of cross-linkage in some area and thus brings about increased resistance to fissure progress. The reason for the increased resistance to fissure progress in a tighter cross-linked and higher oriented duroplastic component can be attributed to higher strength due to a larger number of chemical bonds per volumetric unit. On the other hand, the inhomogeneity of the entire layer, because of the marked differences between the duroplastic and thermoplastic components, may be the reason for the different emission behavior since progressive micro-fissures are occasionally detected at the boundaries between them.

The measurement of acoustic summation curves on adhesive joints of metals offers, therefore, results that are characteristic
for adhesives, permits differentiation between microscopic and macroscopic damage cycles and allows clear observation of changes due to ageing processes.

4.2 Pulse Cams

Conspicuous differences between the adhesives examined can also be determined through the intensities of acoustic emission and the pulse cam distribution. The overall sums of the pulse cams, for the REDUX adhesive for instance, are ten times higher than those for the other adhesives examined. Since the pulse cam is proportional to the root of the pulse energy its size is a direct indication of the energy of the respective failure cycle. The differences mentioned for individual adhesives can be explained by greater elastic energy storage, before the actual progress of the fissure, in REDUX than in adhesive FM 1000, for instance. This energy is released suddenly only during destruction and is partially converted into damage energy. An adhesive layer consisting of REDUX is relatively nonhomogeneous when compared with adhesives FM 1000 and AF 126, which is already recognizable visually. The distribution of polyvinylformal, which is used for plastification, in the duroplastic phenolic resin appears grainy in the fracture planes. This is not the case for the other adhesives. Duroplastic and thermoplastic components are much better mixed in them so that the cured adhesive surfaces present a more homogeneous picture.

The intensity of the released acoustic energy is not a measure for the strength of the bond, however. Much energy can be stored in relatively brittle adhesives like REDUX, while more plastified but more homogeneous adhesives can reduce tension peaks through plastic deformation and thus present greater overall resistance of the adhesive joint to fissure progress.
The analysis of pulse cams may on the other hand convey clear indications of the extent of damage to the adhesive layers if the pulse cam distribution for an adhesive material is recorded for different loads, as shown in Fig. 3, to the left, for adhesive FM 1000 in aged condition for instance. For higher loads the number of higher energy pulses increases. This is to be attributed to the increasing destruction of duroplastic components after corresponding storage of elastic energy through deformation of plastic components.

Fig. 3. Pulse cam distributions of fissure pulses for adhesive FM 1000 (to the left) for a load of (a) 2000 N and (b) 400 N as well as for adhesive AF 126 (to the right) in (a) nonaged and (b) aged condition.

Ageing processes where the, already mentioned, increasing plastification of adhesive materials through influx of humidity takes place, can also be observed by means of the pulse cam analysis, as in Fig. 3 to the right. A higher share of low energy pulses characterizes the aged adhesive as compared to the adhesive in unaged condition. Sometimes that permits the conclusion that even the plastified component is weakened by the entrance of water to such an extent that damage cycles can occur in it; they have no connection, however, to the energy storage before the onset of the fissure.
4.3 Frequency Analysis

Additional possibilities for the identification of recorded fissure pulses are offered, especially by frequency analysis. The spectra of fissure pulses differ clearly from those signals that can be attributed to knocking noises at the sample or to artificially created friction processes, as shown in Fig. 4.

Frequency spectra also convey clear differences in the fissure characteristic of various adhesives in their unaged, as well as aged, condition. The unaged adhesive Redux is characterized by the appearance of two groups of spectra while only one group can be observed for the aged condition. This occurrence can be explained by the great inhomogeneity of the cured adhesive layer since duroplastic and thermoplastic components form their own structural areas here which emit pulses independent of each other during fissuring. Their frequency spectra are clearly different because of the different molecular structures of the phenolic resin (duroplastic) and of the polyvinylformals (thermoplastic). Since apparently only one component emits acoustic pulses in the aged condition, the other must be so highly plastified that its emission is no longer of consequence.

When making an overall comparison between the frequency spectra of the aged adhesive layers and those of the unaged
ones, in Fig. 5, it becomes apparent that the entrance of humidity causes an acoustic emission of higher frequency during the progression of the fissure. With it goes a weakening of the signals of between 5-10 db. At the present state of the study it is not possible to assign this shift of intensities and frequencies indisputably to mechanical cycles of the working materials. As mentioned before, frequency spectra are composed of those of the primary pulses and of the so called transfer function, which describes the damping and oscillating behavior of the adhesive and the joint components. At least the transfer function of the adhesive will change due to ageing processes, meaning primarily the absorption of humidity, to an extent not known until now. On the other hand, the frequency characteristic of the primary system will naturally also change. To be able to derive further changes in the mechanics of the materials from the frequency spectra, the dependence of the transfer function of the adhesive layers on ageing must be found so as to allow subsequent elimination of its influence on the final result. At present it cannot yet be clearly decided how justifiable the necessary effort is to determine the transfer characteristics of all adhesives as a function of their degree of ageing.

The result achieved with the aid of frequency analysis for adhesive AF 126, Fig. 5 lower right, is remarkable for showing that one-day storage of the fissured sample after the climate test, in prestressed condition and with appropriate frequency change, produces the same result as can be measured for the unaged adhesive. Ageing processes in adhesive layers on metals are therefore reversible, at least under the influence of muggy climate and as far as changes in the adhesive are concerned. This was also observed during the torsional-oscillation experiment (6). This reversibility permits the conclusion that ageing processes in muggy climate are to be attributed almost
Fig. 5. Frequency spectra of fissure pulses for adhesives REDUX 775 (above), FM 1000 (below left) and AF 126 (below right)

a: in unaged condition
b: in aged condition
c: (only for AF 126) like b, but 24 h after the climatic test

Investigations of acoustic emission behavior, which were carried out on adhesive metal joints with various adhesives and different preliminary treatments, under mechanical loading in the unaged and aged condition, permit significant conclusions.

The acoustic emission analysis provides, in addition to technological tests of strength, of deformation behavior and of fissure progress in adhesive metal joints, expanded knowledge about the behavior of the adhesive layers under load and climatic influences. In contrast to the
technological tests the acoustic emission analysis conveys insights into the connections between the structure of the adhesive layer and its behavior during mechanical, resp. physical changes.

With respect to their acoustic emission behavior adhesives for metals differ in the unaged and aged conditions, after only brief loading and short climatic exposure, to a far greater extent than is possible to measure in technological tests. For a suitable choice of sample shape ageing periods of a few days without aggravation of environmental conditions are sufficient for characterization of behavior under load and over the long term.

When compared with amplitude and frequency analyses, which require a great deal of equipment, the recording of acoustic summation curves, which can be considered as the simplest procedure of the acoustic emission technology, generally provides sufficient characteristics for the evaluation of the adhesives. The results of acoustic emission measurements are consonant with those that can be obtained with the aid of test methods from fracture mechanics.

Measurements of acoustic summation curves can be made at adhesive bonds before the onset of macroscopic fissures, as well as after the forced damage. The onset of macroscopic fissures is clearly recognizable. This appears to make acoustic emission analysis particularly suitable for the monitoring of components since it permits the recognition of the onset of fractures independent of the size of the bonded system, for instance after stressing to rated load. When a component is used only in the sub-critical range, in which assuredly no macroscopic fissures
originate, differences in deformation and microfissure behavior of the adhesives, such as can be caused by different preliminary surface preparations, can clearly be determined in the unaged condition. Diminution of the pulse sum or absence of acoustic pulses in the subcritical range, as compared to the unaged condition, permits a safe conclusion of soaking cycles in the adhesive layers. An increase of the acoustic sum in the aged condition is, according to the present state of knowledge, caused quite definitely by the generation of macroscopic fissures. The still permissible overall load can therefore be measured relatively simply for aged components.

The acoustic emission analysis is, therefore, suitable for comparative laboratory experiments on adhesives for metals, resp. adhesive joints of metals, for determination of the parameters decisive for strength and reliability, as well as for component testing and quality control during and after the construction of adhesive structures. The use of acoustic emission analysis in laboratory experiments expands the knowledge about the behavior of adhesives for metals under load and during ageing, which has previously been gained mostly through technological tests. The use of acoustic emission analysis permits beyond that a drastic shortening of weather exposure periods, for long-term experiments, for instance. Its application for the bonded component at a subcritical or rated load offers reliable references about the condition of the bonded joints and, perhaps, about macroscopic fracture processes going on in them.

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