Platinum sensors embedded in ESCC-4006/014 modified construction:

A turbulent tale about the coexistence of dissimilar materials

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ABSTRACT

The present article focuses on a failure detected during the qualification testing of a type of RTDs (Resistance Temperature Detector) manufactured by TE Connectivity, radial/epoxy-bead type construction, with a platinum thin film resistor as sensing element. It details the failure analysis, root cause identification, mitigation strategy, validation of the improved product and the lessons learnt.

RTD TYPE UNDER STUDY AND HERITAGE OF USAGE

The RTDs under study are manufactured by TE Connectivity (formerly Measurement Specialties/BetaTherm) as an evolution of their ESCC-QPL (European Space Components Coordination – Qualified Parts List) NTC (Negative Temperature Coefficient) thermistors, produced in accordance to [1], with the aim to improve the temperature acquisition absolute accuracy and the temperature range, in the lower end.

The construction of the ESCC-QPL NTC thermistors is very similar to the one described in [2]. The following three main modifications were conducted in them to evolve into the RTDs under present study.

- The internal NTC temperature sensitive element is substituted by a thin film platinum temperature sensor manufactured by IST AG, company listed in ESCC-QPL for this technology. The new sensor belongs to the same family of products as the ESCC-QPL ones, being the main difference the smaller dimensions in the chosen one. The main advantages brought by the new sensor are the improved absolute accuracy in the temperature acquisition (class B, as per [3]) and the compatibility with wider temperature ranges ([-200, 600]°C).
- The original epoxy material Loctite Stycast 2850FT, used to encapsulate the sensor in a bead, is substituted by 3M[™] Scotch-Weld[™] Epoxy Adhesive EC-2216 B/A Gray. This new epoxy is widely used in space applications, suitable for bonding materials of very different nature in a very wide service temperature range, including cryogenic temperatures.
- The two wires used as electrical interface are changed to the ESCC-QPL ones according to [4], tolerant to lower temperatures and highly resistant to the ionizing radiation.

In Fig. 1 is depicted the main elements that constitute the RTD type under study and details of the ESCC-QPL NTC thermistors internal construction, for comparison purposes.



Fig. 1. Details on the construction of the RTD type under study (three first images from the right); internal construction original ESCC-QPL NTC thermistors

This new type of RTD was specifically developed during the end of the last decade for the ESA (European Space Agency) missions Sentinel-4, ExoMars and Galileo. For these missions, the concrete electrical type needed was a RTD of 1000Ω at 0°C, which was branded by TE connectivity with the part number "PT1000D355". The robustness and reliability of the new RTD was proven through production control, screening and qualification test campaigns based on the flows of tests and methods required by [5]. The tests were tailored in accordance with a target storage temperature range of [-125, 160]°C and an operating temperature range of [-60, 125]°C, in accordance to project needs. Results were satisfactory, though certain setbacks were found, in the first prototyping validations.

DETAILS ON THE LAST PROJECTS USING THE RTDS UNDER STUDY

In the last years, OHB System AG has been contracted for the manufacturing of units and elements embarked in FLEX (FLuorescence Explorer) and PLATO (PLAnetary Transits and Oscillations of stars) ESA's missions.

FLEX mission will provide global maps of vegetation fluorescence that can reflect photosynthetic activity and plant health and stress. FLEX will carry the high-resolution FLORIS (Fluorescence Imaging Spectrometer), which will acquire data in the [500, 780]nm spectral range. Part of FLORIS is a FPS (Focal Plane System). Part of FPS is a thermal control loop for the settling and stabilization of the temperature of the imaging detectors embarked. This thermal control loop requires highly accurate and robust temperature sensors for the temperature acquisition.

PLATO mission has as objective to find and study a large number of extrasolar planetary systems, with emphasis on the properties of terrestrial planets in the habitable zone around solar-like stars. PLATO payload consists of 26 cameras, each of which integrates a FEE (Front-End Electronic) equipment. The external temperature of the FEEs is needed to be monitored, for which is required a moderately accurate and robust temperature sensor.

For these needs, OHB System AG proposed the usage of the RTD "PT1000D355" with the same production control, screening and qualification test flows conducted for the previous missions, as per [5], with a narrower storage temperature range of [-125, 125]°C.

Additionally, it was added the acquisition of eleven "zero power resistance" measurements in the temperature range [-50, -10]°C, after the end of the screening tests (chart F3, as per [5]), on a subset of 23 RTDs to be used in the FPS of FLEX FLORIS. This data was used to calibrate each RTD, i.e., fitting a R=R(T) function for each of them, with the aim of improving the absolute accuracy in the temperature acquisition.

DESCRIPTION OF THE FAILURES FOUND DURING THE QUALIFICATION TESTING

TE Connectivity produced an assembly lot, with DC (date code) 2041, and initial size of 330 pieces at the beginning of the production control tests (chart F2, as per [5]) for FLEX and PLATO missions. Production control and screening tests were completed successfully. Later, 56 pieces were segregated from the screened assembly lot and the qualification tests (chart F4, as per [5]) were initiated with them.

In April 2021, TE Connectivity announced that, after the "thermal shock" test, during the "external visual inspection", failures were detected: the epoxy bead was partially detached from the baseplate on 2 out of 12 RTDs. Fig. 2 shows images of these defects. "Zero power resistance" measurements at 0°C on the affected RTDs were nominal; hence the failures didn't affect the electrical functionality of the RTDs.



Fig. 2. Delaminations detected during the qualification campaign

The temperature extremes, during the "thermal shock" test in the qualification campaign, were -125°C and 125°C; the number of cycles was 10. The failed RTDs were subjected previously, during the production control tests, to a similar "thermal shock" test, but limited to 5 cycles and with temperature extremes -65°C and 125°C. Both "thermal shock" tests were initially planned to be conducted between the same temperatures extremes, but, as per TE Connectivity's request, the lower end during production control tests was limited. The rationale for that request was not to outsource the "thermal shock", during the production control tests, as TE Connectivity's thermal chambers didn't reach -125°C. The outsourcing of any test, during the production control and screening, was not preferred in order to reduce the risk of any damage on the RTDs during the additional transportation and handling operations required. The customers' chain accepted the relaxation taking into consideration that the intended temperature extremes will be used during the qualification tests, the successful heritage data and the same accepted approach in the previous missions.

The qualification campaign continued and the results of the remaining streams were successful.

FIRST ANALYSIS AND RESULTS OF THE INITIAL INVESTIGATION LINES

A major non-conformance was reported to the customers' chain and an NRB (Non-conformance Review Board) was assembled to analyse and resolve the failure of delamination, tasks which required more than one year.

The NRB identified as likely practical cause of the failure the weak adhesion strength between the epoxy bead and the baseplate. The type of failure observed in this study has been for all the cases an adhesive failure meaning an interfacial bond failure has been observed between the adhesive and the adherend.

Interfaces between dissimilar adhering materials can suffer adhesive failure under overstress loads, i.e adhesion failures in bonded joints. The difference in the CTEs (Coefficients of Thermal Expansion) of the epoxy bead and the baseplate, mechanically coupled to expand and shrink together through the adhesion process, induces the built-up of shear mechanical stresses on their interface, during the changes of temperature in each cycle of the "thermal shock" tests. Internal stresses occur in adhesive joints because of a natural tendency of the adhesive to shrink during setting and because of differences in physical properties of adhesive and substrate. The CTE of adhesive and adherend should be as close as possible to minimize the stresses that may develop during thermal cycling or after cooling from an elevated temperature cure. In the present case, the difference in the CTEs of the epoxy bead and the baseplate, mechanically coupled to expand and shrink together through the adhesion process, seems to confirm the idea that the built-up of shear mechanical stresses built at their interface, during the changes of temperature in each cycle of the "thermal shock" tests could lead to the observed bonding failure.

These shear mechanical stresses can lead to the observed adhesion failure, if reaching certain limits. Also, if not reaching such limits, the repetition in their application induces a mechanical fatigue, which weakens the adhesion strength between the interfacing materials, which, ultimately, can provoke the sudden relaxation of the built-up stresses in the next cycle in the form of a detachment. This mechanism could be the cause of the detected delaminations during the "external visual inspection" after the "thermal shock", during the qualification campaign.

However, the epoxy material, EC-2216 B/A Gray, and the baseplate material, aluminium alloy 1000 series, have extensive successful heritage of concurrent usage in harsher conditions than the ones that triggered the detected failure. This indicates that, for these materials, the mutual adhesion strength shouldn't be weak, as evidenced by the failure found that it is.

As such, the NRB discarded the possibility of an erroneous selection of materials in the construction of the failed RTDs. The exact cause of premature adhesive failure is very difficult to determine. An analysis of failure mode, nevertheless, can be an extremely useful tool in determining whether the failure was due to a weak boundary layer or due to improper surface preparation. The NRB concluded that the ultimate root causes of the failure were unknown factors that degraded the optimal theoretical adhesion strength achievable between the affected materials. The identification of these unknown factors, and their mitigation, were essential to resolve the non-conformance and to restore the suitability of this RTD's technology for space applications.

In order to achieve this final goal, the NRB decided to gather empirical evidence of the supposed adhesion strength for this case, besides the delaminations. As such, two initial investigation lines were initiated. In the first one line, using CT (Computed Tomography) scan technique, to investigate the internal characteristics of the RTDs of the failed assembly lot; both using RTDs affected by delaminations and not. In the other line, targeting a quantitative measure of the adhesion strength between the epoxy and the baseplate, bonded using the same manufacturing process followed by TE Connectivity in the failed assembly lot.

Characterization Of The Internal Features Of The RTDs

The 2 failed RTDs during the qualification campaign (serial numbers 2041A296 and 2041A103) and 3 RTDs only screened (serial numbers 2041A106, 2041A165 and 2041A168) were inspected using CT scan technique, focusing in the interface between the epoxy bead and the baseplate. The objective of this comparative investigation was to detect any internal area showing evidences of weaker adhesion, which could be the origin of the delaminations detected externally, propagating from it, due to the accumulation of thermo-mechanical stresses.

In Fig. 3, upper row, it is shown a lateral section of the RTD 2041A296, failed during the qualification campaign. Redframed, it is highlighted the extension to which the external delamination propagates inside the RTD. It can be seen that it reaches a point very close to an internal bubble in the epoxy bead, near the centre of the rougher area of the baseplate (see Fig. 4, for a better view of it), intentionally created to increase its effective contact area, which helps to enhance the adhesion strength of the epoxy on it. Blue-framed, it is highlighted an internal delamination, not seen from the exterior, close to an internal bubble and on the rougher area of the baseplate again.

In Fig. 3, lower row, it is shown two sections of the RTD 2041A168, not showing external delaminations. On the left side, red-framed, an internal delamination can be seen. Besides, it was noted that the shape of the delaminated epoxy surface matches the profile of the confronting baseplate rougher area, except in some circular spots (small voids). This points to that the internally delaminated epoxy, initially, was generally (except the small voids) in intimate contact with the baseplate, to get this profile in its surface, and later the detachment occurred. On the right, red-framed, the same internal delamination can be seen in all its extension, all of it within the rougher area of the baseplate. Also, over this rougher area, a myriad of small voids can be seen between the baseplate and the epoxy bead.

These observations confirmed that the adhesion state between the epoxy bead and the baseplate, even in RTDs not subjected to the qualification campaign stresses, was far from being optimal. Indeed, the same multiple internal features (internal delamination, small voids, epoxy bubbles), correlated to the external delaminations seen in the failed RTDs, were present in RTDs not affected by external delaminations.

Also, the clear correlation between the internal delaminations and voids with the centre rougher area of the baseplates drove the NRB to decide to eliminate this feature on the baseplates in future assembly lots.

Finally, an unexpected finding was observed in the RTD 2041A165, during the "external visual inspection" prior to the CT scan: it showed an external delamination. Indeed, unexpected, as this RTD, at the end of the screening tests, was subjected to an "external visual inspection" by TE Connectivity and this feature was not found.



Fig. 3. Internal characteristics of RTDs; upper row, failed RTD, lower row, regular RTD

During the initial related discussions, the NRB assumed that either it could be a mistake by TE Connectivity in spotting the delamination during the screening or the effect of an unknown environmental agent after the screening, likely during the storage, which caused the delamination.

In order to gather more information, the NRB requested to conduct an "external visual inspection" in the whole set of RTDs of the assembly lot DC 2041 stored at OHB System AG premises. 6 more RTDs affected by delaminations were found. The NRB acknowledged that it would be very unlikely that TE Connectivity would have not detected 7 RTDs with delaminations during the "external visual inspection" by the end of the screening. Hence, the attention was focused in identifying the unknown environmental agent that could explain the appearance of delaminations in RTDs after the screening flow.

During this analysis, the NRB identified a clear correlation between all the 7 RTDs showing external delaminations, not subjected to the qualification campaign: the 7 RTDs were subjected to the extra calibration process required for the 23 RTDs of FLEX FLORIS FPS. The NRB inferred that the additional measurements of the "zero power resistance" at different temperatures could be the cause. These measurements induced the RTDs to temperature changes, which can lead to the propagation of pre-existent internal delaminations, as seen in the CT scan, into the external ones later.

The NRB verified that, unfortunately, after the data acquisition for the extra calibration of the RTDs for FLEX FLORIS FPS, TE Connectivity didn't conduct an "external visual inspection", as this was not explicitly required by OHB System AG. Also, during the "incoming inspection" at OHB System AG, the "external visual inspection" was only required by sampling. Hence, it was possible that these delaminations were already present after the calibration process and were not detected neither by TE Connectivity (not checked) nor by OHB System AG (by chance), being only spotted afterwards during these later investigations.

First Estimations Of The Adhesion Strength Between The Epoxy Bead And Baseplate

In order to get a rough and quick quantitative estimation of the adhesion strength between the epoxy bead and the baseplate of the failed assembly lot DC 2041, there were manufactured several test samples in a simplified, though representative, configuration. Each test sample consisted of two baseplates, bonded through an epoxy droplet in the manner depicted in the Fig. 4, leftmost image. The two baseplates were pulled, subjecting the bonding area to shear stress, until they detach (destructive test till failure). The shear stress to failure was deemed as a good estimator of the sought adhesion strength.



Fig. 4. Test samples geometrical configuration (left) and example of failure of adhesive type (centre and right)

This geometrical configuration of the test samples has as main drawback that the control on the effective contact area between the epoxy droplet and the baseplates is poor: when the two baseplates compress the epoxy droplet, it increases its contact surface with both against them. The effective surface can only be estimated after the detachment, inspecting the shape of the remaining droplet adhered to the baseplates. But, it is difficult to estimate a precise contact area, due to the irregular shape of the droplet. This induces a significant uncertainty in the shear stresses estimated. But, as first rough qualitative and quick estimation of the adhesion strength, it is a suitable technique.

6 samples were manufactured with baseplates as received from their manufacturer. The average measured shear stress to failure was 6,05MPa (standard deviation, 0,65MPa). 6 samples were manufactured with baseplates prepared as the ones used in the failed assembly lot DC 2041. This preparation was an immersion in acetone, at ambient temperature, during 60-120s. The average measured shear stress to failure was 7,52MPa (standard deviation, 1,1MPa).

The measured shear stresses to failure were three times smaller than those expected as per the "Overlap Shear" test according to [6].

Besides, all the detachments were of adhesive type failures between the epoxy and one of the baseplates, which was evidenced by the absence of epoxy residues on the detached baseplate, as shown in the Fig. 4, centre and rightmost images. No cohesive failures occurred in the epoxy or the baseplates.

ROOT CAUSES IDENTIFICATION, MITIGATION STRATEGIES AND PRELIMINARY VALIDATIONS

At this stage, the data collected by the NRB confirmed, qualitatively and quantitatively, the existence of a weak adhesion strength between the epoxy bead and the baseplate in this type of RTDs manufactured by TE Connectivity. This conclusion was not limited to the assembly lot DC 2041, as could only be assured from the data obtained through the CT scans; indeed, the data from the quantitative adhesion strength is representative for any assembly lot manufactured.

In order to identify the hidden root causes that weakened the optimal adhesion strength, as per [6], the NRB investigated which were the recommended conditions of application by the manufacturer of the epoxy EC-2216 B/A Gray.

Generally, the optimal adhesion strength of any structural epoxy depends not only on the epoxy itself, and how it is prepared, applied and treated afterwards. It also strongly depends on the substrate surface state to be bonded to. Particularly, the epoxy EC-2216 B/A Gray when bonded to an aluminium substrate, according to [6], requires a stringent previous preparation of the substrate to achieve an optimal bonding. The objective of this preparation is to eliminate as much contaminants and oxidation as possible from the surface of the substrate and present a freshly pristine surface for bonding. This preparation increases the surface energy of the substrate, making the aluminium atoms of the surface more prone, in order to minimize the surface energy of the substrate, to form tight chemical bonds with the functional groups of the applied epoxy, enhancing the overall chemical adhesion between the epoxy and the aluminium substrate.

TE Connectivity confirmed that the sole preparation applied to the baseplates before the application of the epoxy, in this type of RTDs, was their immersion in acetone, at room temperature, during 60-120s.

The NRB agreed in that this didn't fulfil any of the suggested methods in [6] for the substrate preparation. The expected action of the acetone is limited to clean partially the baseplates: its nature of polar solvent makes it very effective again polar molecular contaminants, but not as much against non-polar molecular contaminants. None action is expected from the acetone on the oxidizing state of the surface of the baseplates.



Fig. 5. ATR-FTIR spectroscopy and droplet test results on baseplates, left column as manufactured, right column acetone cleaned

The effectiveness of the cleaning using acetone was tested through ATR-FTIR (Attenuated Total Reflectance-Fourier Transformed Infrared) Spectroscopy conducted on the surface of the baseplates in two configurations: as received from their manufacturer and after being cleaned with acetone by the procedure of TE Connectivity. As can be seen in the Fig. 5, transmittance valleys near 3000cm⁻¹, associated with absorption wavelengths of certain types of carbon-hydrogen bonds, were found in the two configurations. This indicated the presence of organic contamination in the surface which was not entirely removed after the acetone cleaning.

An additional characterization to confirm the non-optimal state of the baseplates before the epoxy application was conducted using the "water droplet" test, which allows to assess the surface energy of the baseplates. The same two configurations characterized in the ATR-FTIR spectroscopy were assessed. As can be seen in the Fig. 5, the measured contact angles in both configurations were near 90°, which indicates a poor wettability of the surface and, hence, its low surface energy (reduced bonding affinity). Also, the similarity in the measurements in both configurations indicates that the increase in the surface energy induced by the acetone cleaning was negligible.

As per these results, the NRB concluded that the likely ultimate root cause of the poor adhesion strength was an insufficient surface preparation of the baseplates before the application of the epoxy. The NRB discussed the possible improvements to mitigate this root cause. Three main changes were identified, based on the general surface preparation methods for aluminum substrates described in [6]:

- Improving the cleaning of the bonding surface, using solutions of non-polar solvents (hexane and Isopropyl alcohol), polar solvents (acetone) and proprietary alkaline cleaner Sococlean type, from Socomore. To enhance the cleaning actions, usage of elevated temperature and US (Ultra-Sonic) energy.
- De-oxidizing the bonding surface, through mechanical abrasion, fine grit abrasives, and chemical etching, using REACH compatible proprietary acidic etchant Socosurf type, from Socomore.
- Using primers to enhance the adhesion strength and its robustness against the environmental aging. 3M recommends the primer EC-3901 for improving the adhesion on metals for their structural adhesives.

If the identified root cause was correct, the use of different combinations of these improvements should increase significantly the adhesion strength. To confirm this, the NRB agreed in using the same quick and rough method described in the previous section. Test samples were built using baseplates prepared with different combinations of these suggested improvements. The different configurations tested and the measured adhesion strengths are presented in the Table 1.

In all the trials, the measured adhesion strength improved greatly, compared with the one obtained using the surface preparation followed by TE Connectivity up to now. Besides, most of the failure modes were of a mixture cohesive/adhesion type, as shown in Fig. 6 (first row, left side), which is another indicator of a healthier adhesion.

Trial	Cleaning Method	De-Oxidizing Method	Primer EC-3901	Shear stress to failure	
				Average (MPa)	Standard Deviation (MPa)
Ι	Hexane/Isopropyl alcohol+Acetone (15 minutes, US energy and 40°C for both solutions)	None	No	12,55	1,21
II	Hexane/Isopropyl alcohol+Acetone (15 minutes, US energy and 40°C for both solutions)	None	Yes	13,71	1,82
III	Acetone (15 minutes, US energy and 40°C)	Mechanical grinding	No	11,75	2,47
IV	Acetone (15 minutes, US energy and 40°C)	Mechanical grinding	Yes	14,93	3,17
V	Sococlean (15 minutes, US energy and 40°C)	Socosurf (5 minutes, US energy and 40°C)	No	15,93	1,42
VI	Sococlean (15 minutes, US energy and 40°C)	Socosurf (5 minutes, US energy and 40°C)	Yes	20,29	3,60
VII	Hexane/Isopropyl alcohol+Acetone (15 minutes, US energy and 40°C for both solutions)	Socosurf (5 minutes, US energy and 40°C)	Yes	18,52	3,48

Table 1. Results of trials to validate the proposed root cause of the failure and characterize different mitigation means

Results from trials I and II suggest that a more thorough cleaning of the surface helps significantly. Likely eliminating the contamination detected on the surface of the baseplates in the previous section. To verify this, the NRB asked to conduct ATR-FTIR spectroscopy of a baseplate cleaned with hexane. Results are shown in Fig. 6 (first row, right side): the absorption lines detected on baseplates only cleaned with a polar solvent, like acetone, disappeared.

Results from trials III and IV suggest that the elimination of the oxidized surface using mechanical means, alongside any trapped tenacious contamination in it, greatly improves the adhesion strength as well.

Results from trials V to VII suggest an enhanced positive effect in using chemical etching instead of mechanical abrasion for the de-oxidizing of the bonding surface. Also, the improvements brought by the usage of alkaline cleaners, over polar and non-polar solvents, in the cleaning process. As additional reassuring information, "water droplet" test was conducted in baseplates treated as the trial VI. Results are shown in Fig. 6 (second row, left side): the contact angle measured was significantly smaller than the one seen on baseplates only prepared with a polar solvent cleaning, like acetone, which indicates a greatly enhanced surface energy, which benefits the adhesion process.

All the trials showed that the usage of a primer was greatly beneficial as well.

In order to finalize the analysis, the NRB identified the need to characterize the adhesion strength in a more controlled and repeatable manner, eliminating the uncertainties of the method used up to now. For that, the NRB decided to use the "Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to-Metal)" test, according to ASTM D1002. The geometrical configuration of the test specimens, and the test set-up, is shown in Fig. 6 (second row, right side). In light grey, the metal plates, in dark grey, the adhesives used are shown.

10 test specimens were prepared by TE Connectivity in 2 different configurations: 5 specimens whose substrates were prepared as the baseplates in the failed assembly lot DC 2041; 5 specimens whose substrates were prepared with a procedure proposed very similar to the one followed in the best trial (VI) described in Table 1. At ESA's facilities, the test specimens were pulled to failure. The results obtained are provided in the Table 2.

Table 2. Results of final trials to validate accurately the proposed root cause and the most promising mitigation mean

Trial	Cleaning Method	De-Oxidizing Method	Primer EC-3901	Shear stress to failure	
				Average (MPa)	Standard Deviation (MPa)
А	Acetone (1-2 minutes, 25°C, no US energy)	None	No	9,69	0,39
В	Sococlean (15 minutes, US energy and 40°C)	Socosurf (5 minutes, US energy and 40°C)	Yes	18,88	0,57



Fig. 6. Miscellaneous data to validate proposed root cause (first row and second row, left) of the failure and test set-up according to ASTM D1002 (second row, right)

The data collected confirmed an improvement ($\approx x2$) of the adhesion strength using the most promising new surface preparation process (Table 2, trial B) compared to what could have been expected in the failed assembly lot DC 2041 (Table 2, trial A). Also, all the adhesion failures were of cohesive type in the epoxy, which indicates that the cohesive strength of the epoxy was weaker than its adhesion to the baseplates, which is a clear sign of an optimal bonding.

At this stage of the study of the non-conformance, the NRB decided that it had been gathered sufficient information to ascertain the following main conclusions:

- The primary cause of the delaminations found in the assembly lot DC 2041 was the weak adhesion strength between the epoxy bead and the baseplate of the RTDs.
- The ultimate cause of the weak adhesion strength was the unsuitable surface preparation process used on the baseplates.
- These causes are not exclusive of the assembly lot DC 2041, due to any particular mistake or deficiency in its production. They were present in all the manufactured assembly lots of this type of RTDs. However, the fact that these failures were not seen in the previous assembly lots, and their frequency of appearance in DC 2041 was relatively low (<20%), indicates the existence of a variability in the adhesion strengths that can explain the positive results in previous assembly lots just due to the pure statistical reasons.</p>
- It has been identified a suitable set of mitigation means to prepare the surface of the baseplates in a manner in which the adhesion strength improves to limits close to the best achievable. Though, the NRB recognized that this data was limited to the adhesion strength at the beginning-of-life of the bonding. No data was collected on the evolution of the adhesion strength after the effects of aging environmental factors. The NRB decided, based on criteria of schedule and confidence, to produce this data during the qualification campaigns of future assembly lots.

Supported in these conclusions, the NRB agreed in that the non-conformance review process had progressed sufficiently to consider the release of the production of new RTDs for FLEX and PLATO missions, after some final discussions, summarized in the next section, would have been addressed.

UNVEILING A HIDDEN UNREALISTIC STRESS DURING THE SCREENING TESTS

Looking back to the correlation found during the CT scans, the NRB was concerned by the evidence that the "zero power resistance" measurements during the calibration induce a wear-out in the RTDs. These measurements were made in the same conditions as the ones conducted during the screening tests, within the operating temperature range of the RTDs. The electrical measurements conducted during the screening flow don't intend to add any wear-out to the RTDs, but just spotting outliers in the assembly lot that cannot bear the actual induced stresses applied ("thermal shock" and "burn-in" tests).

Nevertheless, the explanation for this correlation may just simply be that the overall quality of the assembly lot DC 2041 was so poor that the weakest of their thermistors could be sensitive to the fatigue of the repetition of the thermomechanical stresses induced by the "zero power resistance" measurement, whilst a healthy assembly lot wouldn't be.

However, in order not to overlook any uncontrolled factor, the NRB asked TE Connectivity to detail how the "zero power resistance" measurements were conducted for these RTDs. The manufacturer explained that the methodology was exactly the same as followed for measuring this characteristic in their ESCC-QPL thermistors. As per the requirements of [1] §8.3.1.1, the chosen *controlled uniform medium capable of maintaining an accuracy of* $\pm 0,01^{\circ}C$ was a liquid thermal medium, cooled or heated to the desired temperature for the measurement. Once the liquid thermal medium reached stably the desired temperature, the RTD to be measured, held at ambient temperature until that moment, was immersed in the liquid. Then, the "zero power resistance" of the RTD was continuously monitored and, when it reached a stable value within the accuracy of the measurement equipment, the thermal equilibrium between the RTD and the liquid thermal medium was achieved. At this moment of time, the "zero power resistance" measured was noted.

The NRB recognized that this immersion process was essentially a "thermal shock" test, as described in MIL-STD-202, Test Method 107, using liquid baths. This type of "thermal shock" is more severe than the one applied during production control and qualification tests, using gaseous mediums, due to the quicker rate of temperature change. This is a severe stress that can easily explain why it added, on its own, a great deal of thermo-mechanical fatigue to the RTDs during the "zero power resistance" measurements, in the frame of the calibration process, but also during the production control, screening and qualification tests.

TE Connectivity recognized so, but argued that this procedure was followed during decades for the ESCC-QPL thermistors with none reliability impact. The NRB acknowledged so, but also pointed to that the epoxy used in the ESCC-QPL was different (Loctite Stycast 2850FT) and, as proven during the initial ESCC qualification and later periodic lot acceptance tests, its adhesion strength was sufficient to cope with these stresses. However, there is not the same amount of data that proves the same when using the EC-2216 B/A Gray epoxy, at least as applied up to now.

To validate the hypothesis of the NRB, 12 RTDs from the failed assembly lot DC 2041, not subjected of the additional measurements for the calibration required in FLEX mission and to the qualification campaign, were selected. The RTDs were immersed in a liquid thermal medium at -60°C for 4 minutes. After removal from the liquid medium, 4 minutes at ambient temperature were waited. This selected time, as per the "thermal time constant" of these RTDs, allowed them to reach the thermal equilibrium, with both the ambient and the liquid thermal medium at -60°C. This process was asked to be repeated 40 times.

4 RTDs failed, showing the same type of delaminations depicted in Fig. 2. The first one failed only after 5 cycles (the rest after 10, 30 and 40 cycles). The "zero power resistances" of the RTDs at 0°C were compliant despite of the delaminations.

The information gathered through this characterization was of great usefulness. The following main conclusions were extracted from it:

- It was possible to reproduce the failure mechanism that triggered all the activities of the NRB, which reassures the correct understanding of what happened, and why, in the failed assembly lot DC 2041.
- The used method to reproduce the failure mechanism serves, as well, as a valid destructive test method to spot weak adhesion strength issues in future assembly lots. Indeed, if implemented as part of the first steps of the production control flow of the new assembly lots, it could help to reassure the proper adhesion strength, reducing greatly the possibility of failures during the rest of production control, screening and qualification tests. This is an important advantage to save time and resources on defective assembly lots.
- It proved the existence of a hidden, potentially destructive, thermal stress induced during the "zero power resistance" measurements, conducted at multiple stages of the production control, screening and qualification of this type of RTDs. This type of stress is not representative of any application condition foreseen. Therefore, the NRB decided to change the methodology for these measurements to eliminate it.

The NRB decided to integrate the identified new processes in the production of future assembly lots for FLEX and PLATO missions.

PRODUCTION OF THE NEW ASSEMBLY LOTS AND QUALIFICATION RESULTS

At this stage, the NRB decided to launch the production of new assembly lots for FLEX and PLATO missions. The following main modifications were implemented in the production processes of the RTDs and their production control,

screening and qualification tests flows by TE Connectivity. These modifications reflect the practical lessons learnt during the detected non-conformance resolution process.

- The centre rougher area was removed from the baseplates used in new assembly lots of this RTD type for space applications.
- Prior to the epoxy application, the baseplates were prepared following a process based on the characterized in the trials B (Table 2) and VI (Table 1). After the chemical etching step, using Socomore's Socosurf agent, a "water droplet" test was introduced on two samples in order to check the suitable surface energy of the baseplates.
- In the production control flow, after the "encapsulation" step, it was introduced a new destructive test on 10 samples randomly selected: a "liquid thermal shock" test, alike to the one described in the previous section, capable of spotting very early weak assembly lots regarding the adhesion between the epoxy beads and the baseplates.
- The procedure to measure the "zero power resistance" was modified to minimize any thermo-mechanical stresses. The main modification introduced consisted in a pre-heating/cooling stage of the RTDs before being immersed in the thermal liquid medium. As such, initially the RTDs are introduced in a thermal chamber where, with a relatively slow rate of temperature change, are heated/cooled to a close temperature to the one set in the thermal liquid medium. Afterwards, the RTDs are removed from the chamber and immersed in the liquid thermal medium, minimizing the transfer time. This way, the thermal shock experienced by the RTDs during the immersion was practically vanished.
- The storage temperature range was narrowed to the worst-case expected in FLEX FLORIS FPS and PLATO Payload FEEs applications: [-95, 125]°C. The "thermal shocks" during the production control and qualification flows were conducted accordingly. No exemption was accepted anymore in the production control flow, hence outsourcing of the activity was required.
- Additional "zero power resistance" measurements, for calibration purposes, if needed, were embedded within the "High and Low Temperatures Electrical Measurements" conducted during the screening flow, before the final "external visual inspection".

A first tentative small lot was built for FLEX FLORIS FPS, size 97 pieces. Production control and screening tests flows were conducted, as per [5]. Results were compliant with the requirements. Full qualification campaign, as per [5], was conducted. No failures were found.

A second large lot was built for PLATO Payload FEEs, lot size 309 pieces. Production control and screening tests flows were conducted, as per [5]. Results were compliant with the requirements. Partial qualification campaign, limited to the streams associated with thermal stresses, as per [5], is on-going. Though, no failures were found up to the time of writing this article, which included the consecution of the tests that triggered the delaminations in previous assembly lots.

These successful results proved that the non-conformance was resolved satisfactorily for FLEX and PLATO missions and that the new assembly lots comply with the demanded requirements for proving their space application worthiness. Generally, these results restored the confidence in that this type of RTDs can be still considered for applications in the space environment in future missions naturally.

Later on, ESA and OHB System AG have procured additional assembly lots for new needs and the gathered results, up to now, were satisfactory.

LESSONS LEARNT

The benefits of replicating the failure mechanisms and understanding their underlying root cause

The delaminations found in the assembly lot DC 2041 (Fig. 2) were not the first time seen for this type of RTDs. As already mentioned during the description of the previous heritage of use of this type of RTDs, in the first prototyping, *circa* 2015, some setbacks were encountered during the qualification campaign: these very same delaminations.

Back then, during the failure analysis activities, the NRB assembled focused as well in the interface between the epoxy bead and the baseplates. The appropriate cleaning of the baseplates was investigated. At that time, the baseplates were cleaned only with isopropyl alcohol, one of the options recommended for aluminium surfaces, as per [6]. This was the same procedure as followed for the ESCC-QPL thermistors, as per [1], kept unaltered despite the change in the epoxy type for the bead. Besides, the NRB questioned the proper control on the test that triggered the failure ("moisture resistance" test, as per [5]), conducted at TE Connectivity's facilities in the United States of America, which could have induced an unintended overstress on the test objects.

The NRB recommended to change the cleaning agent, using instead another of the possible options as per [6]: acetone. Also, to conduct the "moisture resistance" test in an independent and recognized test house in Europe. ALTER Technology France (formerly, HIREX) was chosen.

Two sets of new test specimens were prepared: 5 with baseplates cleaned with isopropyl alcohol and 5 with acetone. The qualification campaign results were positive in both, no more delaminations were found. This suggested that the cleaning process was not the origin of the issue, though it was decided to ask TE Connectivity to use acetone in the future. Instead, it was considered as the root cause an unknown error occurred during the execution of the "moisture resistance" test, which caused the delaminations.

Retrospectively, with the gathered knowledge summarized in this article, it is clear that the root cause then was not a mistake in the execution of a test, but the same as found for the assembly lot DC 2041.

The authors of this article consider that the main criticism that can be pointed to how this non-conformance was treated in the past is that the NRB didn't identify the error made during the "moisture resistance" test and didn't reproduce it, in order to replicate the failure mechanism. In fairness, it must be said that this *weakness* was already noted in the nonconformance report, as highlighted by a member of the NRB during one of the meetings (Denis Lacombe, ESA), and recognized by the rest of the board. However, it was agreed to assume the risk and close the NRB.

If the identification of the actual error made during the "moisture resistance" test would have been pursued back then, very likely, it would have been concluded that this was not the utter root cause and further investigations would have been demanded. This would have required more time and resources to resolve the problem then. But, doing so, it would have made the overall time and resources invested in that NRB worthy (actually, they were wasted, retrospectively) and would have avoided the failure in the assembly lot DC 2041, as well as the product assurance uncertainties created in any assembly lots produced in-between, despite the successful tests campaigns.

One lesson learned is the recommendation to pursue the basic principles of identifying the root cause of the failures and replicating them as an actual economical practice in the long-term, despite in the short-term, for the specific needs of the affected project, immediate positive results at the end of a testing flow are *sufficient* to justify the use as-is.

Strengthening of the collaboration between EEE parts engineers and MMPP engineers

Materials, Mechanical Parts and Processes (MMPP) engineering ensures the suitability of all the materials and mechanical parts, as well as used processes, in the manufacturing of equipment for the space segment. On April 2021, when the failure of the assembly lot DC 2041 was announced, during the first discussions on how to handle it, the PA management team of the customers' chain recognized that the nature of the failure was utterly related with an adhesion process, for which MMPP engineering has extensive knowledge and experience from the processes involved in the manufacturing of equipment. As such, MMPP engineers were required to be part of the NRB since the beginning. This approach is not customary, as non-conformances related with EEE parts are mainly addressed by EEE parts engineers and radiation engineers only.

Hindsight, this decision was the key difference to the organization in previous NRB, dealing with the failure in the prototyping phase, which brought a more positive outcome in the non-conformance resolution this time. Indeed, the MMPP engineers were the leading voice in the NRB to propose the methodology to confirm the poor adhesion strength, to identify the deficiencies in the processes of preparation of the baseplates and to point to the right mitigation strategies.

The authors of this article consider that the positive experience of this approach has a great general value to be considered for the future. The manufacturing of any EEE part involves materials, mechanical parts and processes. It is logical to consider that the contribution of the MMPP engineers can be useful to discuss failures of EEE parts whose cause can be suspected as related with materials, finishes, processes, etc. As well, as part of the evaluation of new EEE parts, there are several activities related with the assurance of their robustness and reliability dealing with how they are designed and manufactured (e.g., "constructional analysis", audit in the manufacturing line, etc.); the authors of this article consider that the involvement of MMPP engineers in these tasks is utterly useful to spot deficiencies and mistakes at this stage, saving a great deal of time and resources discarding intrinsically weak EEE parts.

Hence, the authors of this article recommend a strengthening in the collaboration between the EEE parts and MMPP engineers in these concrete aspects mentioned and others, in which their mutual disciplines overlap.

Proposal to consider changes in the testing flows of similar technologies

The experience with this type of RTDs is that, having proven that their construction was intrinsically non-optimal regarding the adhesion of the epoxy bead with the baseplate, the empirical results in the majority of the produced assembly lots have been successful. This points to that the dispersion in the robustness characteristics of these RTDs, within each assembly lot and between them, was significant. Also, to that the worst-case robustness RTDs were close to the limits of the stresses exerted during the production control, screening and qualification campaigns, as defined in [5]; in cases, below them, like during the failures experienced, but not always, like in the successful lots.

In this aspect, based on the described methodologies here-in, the authors of this article recommend the consideration of including the "thermal shock" test, using liquid baths, as per [7], as a mean to overstress destructively this type of construction, or similar (where intimate mechanical coupling between dissimilar materials exists) and trigger failures. This test will provide information on the thermo-mechanical robustness of the EEE parts (number of cycles to failure) and its variability among the sampling size. High variability and low worst-case robustness will indicate inhomogeneous quality and likelihood of failures during the regular tests during production control, screening and qualification flows. This test can be considered as part of the first component evaluation of new passive EEE parts candidate for space applications. Later, as well, it can be used as a new element in the regular production control tests flow, executed on a sample and of destructive nature, for the same purposes and to control the variabilities between assembly lots as well, comparing with previous acquired data.

Besides, the authors of this article encourage the community a critical reading of the test methods used during the production control, screening and qualification flows of passive EEE parts. The experience gathered in the study of this non-conformance proves that, in many of these methods, there are defined required environmental conditions to be met, but not precisely how to reach them, which could be a source of unintended thermal, mechanical, radiation and reliability stresses. In these cases, the authors recommend the community to request to the manufacturer of the part a detailed description on how the method is executed, step-by-step, in order to judge if there is a potential hidden stress to be controlled or avoided (e.g., in our case, instead of controlling the temperature with liquid bath, doing so with Peltier cells). Finally, all the detailed information should be part of the procurement specification to ensure its compliance.

REFERENCES

- [1] European Space Components Coordination, "Thermistors (thermally sensitive resistors), NTC, range 2000 to 100000 Ohms at +25°C with a temperature range of -60°C to 160°C," *ESCC-4006/014*, issue 10, December 2013.
- [2] United States of America Department of Defense, "Destructive Physical Analysis for Electronic, Electromagnetic and Electromechanical Parts", *MIL-STD-1580*, issue C, 31 October 2019, figure 20-1.
- [3] International Electrotechnical Commission, "Industrial platinum resistance thermometers and platinum temperature sensors", *IEC-60751*, edition 3, 27 January 2022.
- [4] European Space Components Coordination, "Polyimide Insulated Wires and Cables, Low Frequency, 600V, -200 to 200°C," *ESCC-3901/019*, issue 3, May 2013.
- [5] European Space Components Coordination, "Thermistors (thermally sensitive resistors)," *ESCC-4006*, issue 4, January 2018.
- [6] 3M Company, "3M Scotch-Weld Epoxy Adhesive 2216B/A," Technical Data Sheet, February 2016.
- [7] United States of America Department of Defense, "Test Method Standard Electronic and Electrical Component Parts Method 107, Thermal Shock", *MIL-STD-202*, issue H, 18 April 2015.