EMRP IND13 D2.2.4

Good Practice Guide on "Optimising the thermal stability of joints"

1. Focus of Guideline

This guideline is written for stakeholders that are concerned with or plan to optimize the thermal stability of joints/connections applied in high-precision engineering equipment. It aims to provide information of advantages and disadvantages of joining techniques. It gives hints for choosing the suiting approach and shows their limits of application and the achieved stability of the resulting connection with respect to thermal and also humidity conditions. The guideline contains a comprehension of the measurement results obtained from representative joints which were given in the EMRP IND13 WP2 project report (D2.2.1 – D2.2.3).

The interferometric measurements required high parallelism and flatness of measurement surfaces, therefore, the joints were made of gauge blocks. In conjunction with appropriate techniques, it was possible to achieve a precision of interferometric length measurements of about one nanometre. The orientation (angles in both lateral directions) was determined from linear fitting at certain positions within the phase topography. A typical uncertainty for the angle measurement was estimated to ±0.2" or below. The temperature was varied between (10 ... 50) °C, and the measured lengths were used for CTE determination. To test for thermal stability: length and angles were measured at 20 °C before and after thermal treatment.

Most of the interferometric measurements were carried out in vacuum, therefore, attention was also paid to the behaviour of joints under vacuum conditions. Essential information about the specific geometry and the materials used for the manufactured specimens is given in the text, which is necessary in order to qualify any conclusions derived from the measurement results. The representative joints cover only a limited choice out of the much wider field of joining techniques applied in precision engineering, and joints with different parameters may show different stability properties.

2. Instability Factors that emerge in the nanometric Range

The intrinsic stability of a material can be affected by the relaxation of internal stresses or internal structural changes. Such changes are often the result of external events like thermal (or mechanical) loading, as commonly existent in an industrial environment. An undesirable consequence is thermally-induced drift. First of all, **fabrication procedures** often affect the stability level of a component:

- cutting, drilling or surface grinding can create internal stress;
- thermal treatment can lead to residual strain after cooling;
- thermal treatment can also lead to the transformation between crystalline or amorphous configurations (with specific thermal expansion), while the material is not in thermodynamic equilibrium.

It is noticed that the thermodynamic equilibrium depends on the temperature level. The time it takes for materials to approach equilibrium in which they possess a defined volume varies significantly. A larger relaxation time will affect the thermal stability and make the thermal expansion less predictable.

With growing demands on the accuracy of machine tools, semiconductor device manufacturing, scanning probe microscopy or various assemblies of optical components in ultrahigh-precision instruments, there is a need to investigate not only the stability of certain materials they are made of but also of connections (joints). Generally, a joint consists of different materials with different thermal expansion, interacting with each other at a common interface or interlayer. The overall behaviour of a connection is not only the sum of the behaviours of the isolated parts but also of their interaction. Preferably, the mutual interaction should be rigid or elastic, but also stick/slip friction or penetration, creep and cracking may occur. Connecting agents, like screws or glue, can cause relative movements which are not straightforwardly predictable. Unwanted dust or dirt as well as lubricants can also be a cause of instability.

3. Material Considerations and Wringing of Gauge Blocks

Most of the joints consisted of hardened steel gauge blocks (GBs). Steel is a common construction material with high stiffness and can be very hard. One disadvantage of steel is the relatively high coefficient of thermal expansion (CTE) value of $(11.5 \pm 1) \times 10^{-6}\ K^{-1}$ at 20 °C. Furthermore, if the temperature is raised by more than about 10 K above room temperature, thermally-induced drift can occur even in GB steel. Stacks of each two GBs were subjected to a temperature variation between (10 … 40) °C in the interferometer. Both stacks, measured again at 20 °C, showed a relative length increase of 2.5×10^{-7} which relaxed to the original length during the following 2-3 weeks with an average relative drift rate of $\approx -10^{-8}$ per day.

Because the stability of a joint is also determined by the parts' material, more stable materials like carbides, silicon or SiO_2 should be preferred. However, the stability of a connection may depend on the materials combination, also for different reasons than the thermal stability of an individual material – therefore the choice of material should be made with respect to the specific application. One example is the prevention of thermal strain by using CTE-adapted materials.

4. Screwing (Bolting) Joints

Two holes of 3 mm diameter were eroded into each hardened steel GB or platen. The parts investigated in the stability measurements were bolted with two M3 screws to each other, and a torque of about 1 Nm was applied. The missing of threads in the GBs has the advantage that no bumps are created at the contact surface. **It is important that parts as well as screws, crew nuts and washers are perfectly clean and fit smoothly into each other.** Any dust or sliding of frictional surfaces could lead to time-dependent stress relaxation. And the release of internal stress would cause dimensional changes. The overcoming of critical frictional forces or internal stresses (e.g. yielding) can also be triggered by temperature variations or impact loading.

As a result, no longitudinal or lateral drift was detected at 20 °C during more than one year, also the angles in all directions remained constant within measurement uncertainty. Additionally, the temperature was cycled between (10 ... 30) °C. This did not cause any residual change. The CTE of the connections showed values not different from those for typical GB steel.

5. Adhesive Joints

Two 12.5 mm steel GBs were *longitudinally* glued by a (8.2 ±0.4) µm layer of 2-component epoxy *UHU+ Endfest300* at 20 °C (with CTE_{EP} = 90×10^{-6} K^{-1}), which results in a nominal CTE increase of only 3×10^{-8} K^{-1} according to the epoxy contribution.

A length reduction by 25 nm (-0.3 % of the layer thickness, probably due to epoxy curing) was detected during 100 days, while 90 % of the shrinking took place within the first 50 days. A following heat-treatment at 50 °C for 3 days resulted in a length increase by 10 nm (at 20 °C) followed by a relaxation by -16 nm during another 170 days. It could, however, not be discriminated, if the effect is caused by an instability of the epoxy or of the GB steel. At t = 270 d, the total length reduction was 31 nm. The following apparent length increase may be the result of moisture swelling of the epoxy, because the specimen was measured and stored in air with relative humidity of (40 … 60) % so that moisture might constantly diffuse into the glue joint. No tilting was detected, which is expected due to the small adhesive layer thickness.

Though a thinner adhesive layer was desirable for higher dimensional stability, the achievable thickness is limited by the applicable pressure. A rheological calculation yields the relation for the force needed to reduce the gap distance h by a certain velocity: $F = \frac{A^2}{2h^3}\eta\dot{h}$. For an approximation of the achievable thickness under a certain pressure we can set $\dot{h} \approx h/t_{\mathrm{cure}}$, which yields: $h \approx A\sqrt{\eta/2Ft_{\mathrm{cure}}}$. The adhesive viscosity (which rises during the curing process and tends to infinity when a percolated network of chemical cross links forms) is in the order of $\eta \approx 100$ Pa s, the curing time $t_{\mathrm{cure}} \approx 10{,}000$ s, and the surface area $A \approx 3$ cm^2, therefore, **with a compressive force of *F* = 20 N, the adhesive layer in a "sandwich" connection would not become thinner than about 5 µm.** For a reduction of the drift by a factor 1/10 the force had to be increased to 2 kN (handling a mass of ≈ 200 kg). Also a temperature increase is not a solution, because it does not only lead to a decrease of η but also of t_{cure}.

Two steel GBs were also *laterally* joined at 20 °C by an epoxy layer (average thickness of d = 74 µm). In contrast to numerical results of an FEM model with a parallel adhesive layer, the joint showed significant changes of length and orientation. It was concluded that this is because the adhesive layer was not parallel but varies in thickness by d_{max} - d_{min} ≈ 33 µm along the direction of investigation. A length relaxation in the order of -10 nm was measured, probably due to curing. The specimen also shows a reversible change of the tilt angle between the GBs of ≈ -3" due to a temperature change by 30 K. After returning to 20 °C a permanent tilting by -1" and a lateral movement of the connection by (+30 … +40) nm remained. Although the reason for this behaviour is not clear, the two results are consistent with each other: the thickness variation not only leads to a change of the respective tilt angle when the adhesive volume changes – it also leads to a lateral movement of the connection as a result of the tilting.

Another source of instability was observed which is probably caused by the epoxy outgassing in vacuum and promoted by the increased temperature: after heat treatment at 50 °C a non-uniform adsorption layer on the glued GB measurement face had formed originating from squeezed-out epoxy. After the layer was removed, the measured orientation of the GB changed by +1".

At last, there are several effects influencing the stability in the lateral direction. However, **the large influence of the wedge angle on the dimensional and thermal stability hints to the importance of producing a very symmetric and uniform glue distribution**.

In order to improve the stability, *spacers* were used to produce a hard contact between the GBs. 10 Vol.% of *spherical glass beads* (soda-lime glass, *HELIOS Optics*) were added to the epoxy. The glass beads had a nominal size distribution between (0 ... 50) µm, as given by the manufacturer. The adhesive layer had a final thickness of (70.6 ±0.3) µm, which approximately coincides with the largest glass beads observed under the microscope. As with the pure epoxy, a curing relaxation by nearly -100 nm was observed during ≈ 50 days, but followed by an accelerated length increase (probably moisture swelling) by ≈ +80 nm during 400 days. This is much larger than of the pure epoxy joint, probably because of the much larger glue gap. After 400 d, a saturation seems to take place. Also a glue gap of only 13 µm thickness was produced using smaller glass beads (0 ... 20) µm and additionally a compressive force of about 100 N between the parts in an attempt to crush larger beads, but with similar results as before. This means that **glass beads do not appear to be suitable as stabilizing spacers. If a larger number of spacers shall contribute to dimensional stability, these should not only be as small as possible, but their dimensions should not vary by much more than the allowed length change.** Moreover, these measurements show a significant tilting of up to 2". This could also be explained by moisture swelling of the epoxy. Because the joint is not fully symmetric and some adhesive was squeezed out forming a blob, one side might absorb more water leading to tilting. The CTE (20 °C) of the joint is (13.13 ±0.02) × 10^{-6} K^{-1}, and therefore ≈ 1.5×10^{-6} K^{-1} larger than that of GB steel, most likely influenced by squeezed-out glue.

A much more stable adhesive joint was produced by applying a dilute solution of *synthetic resin* to one GB end face. The thin adhesive layer of $d < 1$ µm was melted together with the joined parts at 150 °C. Two 15 mm silicon single crystal GBs were longitudinally joined in this way. Length, as well as angles, stayed constant during 150 days of observation. A temperature raise to 30 °C for one day resulted in a permanent length increase of +4 nm at 20 °C. Afterwards, the specimen was investigated for about 300 days and showed no further instability. However, this technique is only applicable to thermally stable materials. Steel GBs joined in the same way revealed a relaxation of their lengths after the heating, beginning with a thermally-induced drift rate of ≈ -10^{-8} per day and an estimated total duration of many years.

A hard contact between the end faces of silicon GBs was approached without adhesive in between. A band (≈1 mm thick) of low-outgassing epoxy *Torr Seal TS10* is only acting on the periphery of the contact interface. (In order to avoid a wringing connection between the GBs, one silicon surface had been grinded with an oil pulp of diamonds with 9 µm nominal diameter.) The total measured length relaxation due to epoxy curing at 20 °C in vacuum is only a few nm. After taking the joint out of the interferometer, the length increased by +12 nm, probably

due to moisture swelling, and stays constant after 40 d. Because there is no epoxy trapped between surfaces, the diffusion of moisture only takes a short time to reach equilibrium. The epoxy acting on the sides will create internal strain in the order of -1 % due to curing, while a temperature change by 10 K will only cause a strain of ≈ 0.1 %. Since the moisture also causes a volume increase, it is possible that the parts may separate. Therefore, epoxies with higher shrinkage (dimensional instability) could even be preferred for an optimized thermal stability of the joint. The measured thermal expansion of the joint between (10 ... 40) °C follows that of the silicon parts. As a result of the higher temperature and vacuum, the joint length had reduced by -30 nm. After storage in air it increased again by >20 nm, approaching the length before the thermal cycle. It was concluded that the thermally-induced instability is caused by moisture driven-out at 40 °C, and that the joint length depends on temperature as well as on humidity.

In conclusion: In cases where adhesives like epoxy (that harden by curing or absorb moisture) have to be applied between the parts, the layer thickness should be minimized and adhesive blobs avoided by using less adhesive. Attention should also be paid to the symmetry of the layer, outflow of adhesive blobs can attract significantly more moisture and lead to tilting. In vacuum, epoxy with low outgassing, like *Loctite Hysol*, also known as *Torr Seal*, should be used. A hard contact between the parts without a glue gap can be helpful, when epoxy acting on the periphery of the contact interface is protected against humidity. Furthermore, it should be noted that the influence of the epoxy on the total stability decreases with decreasing amount of adhesive and increasing stiffness of the joined parts. An alternative is to use optical cements that do not need curing: a dilute solution of synthetic resin can even be used to achieve a layer thickness of ≈ 100 nm. However, these are thermoplastic polymers that need to be melted at higher temperature - therefore, the parts have to be made of thermally stable material (cf. 3.).

6. Silicatic Bonding Joints

Connections between fused silica mirror plates (bottom plate: 25 mm, top plate: 10 mm diameters) were manufactured at Fraunhofer IOF, Jena, using advanced joining techniques: silicatic bonding and soldering techniques. These are associated with thermal processes.

The top cylindrical parallel plates have a height l = 5 mm and were joined by a thin SiO_2 chemical layer of $d \approx 100$ nm (silicatic bonding) onto the respective bottom plates. Two joints were measured repeatedly at 20 °C for nearly one year. During the first 100 days both specimens showed a length reduction of the step height by ≈ -3 nm. From the literature (referenced in the articles 3. and 5. in section 8) the length change of the top fused silica cylinder should stay below 1 nm in that time period. A possible reason could therefore originate from the bonding process. A heat treatment is utilized to drive out water. But, some water may resist and could possibly evaporate during the measurements (performed under vacuum) leading to a small volume/length decrease. After 100 days no significant length change was measured anymore. No change of orientation was detected. Also a thermal cycle was carried out at one specimen, and measurements between (10 … 40) °C were performed. Length and orientation of the sample connection at 20 °C showed to be insensitive to a temporary variation of the

temperature. **The high stability of the silicatic bonding joints (with a measured total relaxation of only -3 nm) is mainly due to the very thin bonding layer. An additional advantage of using inorganic silicatic bonding to assemble optical components, compared to using resins (polymers), is the insensitivity to laser radiation.**

7. Advanced Soldering Techniques

For thin-film soldering, a eutectic 80Au20Sn alloy with an average film thickness of (8.9 ±0.5) µm was deposited on the base mirror plate. The solder was melted by a laser spot. The solder film material has a CTE of 16×10^{-6} K^{-1} at 20 °C, therefore, the film is expected to give a contribution to the total CTE of ≈ 0.03×10^{-6} K^{-1}. However, the measured CTE of the thin film-soldered connection is 0.1×10^{-6} K^{-1} higher than that of the silicatic bonding specimen. The three thin-film soldering specimens investigated showed length increases between (+1 … +5) nm during 400 days of measurement. No change of orientation was detected, and also the length at 20 °C was not significantly influenced by the thermal cycling (carried out as above).

Two joints produced by Solderjet Bumping were investigated. Three patches of solder bumps were placed between each pair of cylindrical plates and are bridging a gap of ≈ 100 µm between the plates. It is noteworthy that this joining technology allows, in contrast to the ones mentioned before, for variable, large joining gaps and six DOF alignment. The $Sn_3Ag_{0.5}Cu$ alloy of the bumps has a CTE of 22×10^{-6} K^{-1} and should give a contribution to the total CTE of 0.43×10^{-6} K^{-1}. However, the measured CTE is 1.0×10^{-6} K^{-1} higher than that of the silicatic bonding specimen, because the solder bumps (placed at the lower rim of the top cylinder) are thicker than the gap distance and, respectively, cause a larger thermal displacement.

A specimen that was thermally-cycled between (10 … 40) °C at the beginning showed an initial reduction by -3 nm a total length increase by ≈ +30 nm. A tilting by 0.4” was measured, directly related to the thermal cycling, and another 0.5” during the following 370 days. Another specimen which was not heat-treated in the first place showed a length increase by +10 nm and a tilting by 0.5” during 270 days. Both specimens showed a slowing down of the drift rate (relaxation). To see if a temperature increase accelerates the drift, when the 2nd specimen had a drift rate of only (0.02 ±0.01) nm/d after 300 days, it was heated to 40 °C for 2 days. This resulted in an immediate length reduction by –6 nm and a faster drift of 0.08 nm/d afterwards.

It can only be speculated about the cause of the instability: Because the cooling of the solder happens very fast, the inter-metallic phases are not in thermodynamic equilibrium, which apparently leads to a volume increase. The observations may also be explained by creeping of the relatively large solder bumps under relaxing residual stress (after solidification high thermal stress builds up and overcomes the yield stress, and a rest remains after cooling of the solder) and thermal stress (different CTEs of solder and fused silica may lead to relatively high stress adding to the residual stress or even to fatigue under repeated thermal loading). The relaxation of residual stress should be accompanied by a volume decrease, but the constraints from the cylinders to the solder may also relax and lead to an effective length increase.

It is noted that also the gold-containing thin-film soldering alloy is not a very stable material, partly because of the diffusion of gold atoms. The laser-based soldering process takes

place under very high heating and cooling rates. Therefore, the solidified solder may be far from equilibrium.

In conclusion, the relatively high stability of the thin-film soldering joints (< +5 nm per year) is mainly due to the small thickness of the solder layer, rather than the use of eutectic AuSn alloy. While the Solderjet Bumping technique allows for a higher flexibility in the relative adjustment of the parts, the larger solder gap results in a dimensional instability of up to +30 nm during the first year and a slightly increased CTE of the joint.

8. Comments

Additional information can be found in the following EMRP IND13 related publications:

1. R. Schödel, *Ultra-high accuracy thermal expansion measurements with PTB's Precision Interferometer*. Meas. Sci. Technol. **19**, 084003 (2008)
2. H. Lorenz, R. Schödel: *Interferometric characterization of dimensional and thermal stability of materials and joints*. Proceedings of the 14th euspen International Conference, 02. – 06. Juni, Dubrovnik, Croatia, Vol. 1, pp. 301-304 (2014)
3. H. Lorenz, R. Schödel: *Interferometric measurement of dimensional and thermal stability of joints*. Proceedings of the SPIE, Vol. **9173**, Instrumentation, Metrology, and Standards for Nanomanufacturing VIII, 91730B (2014)
4. H. Lorenz, R. Schödel: *Absolute Interferometric measurement of the dimensional and thermal stability of joining techniques*. 58th Ilmenau Scientific Colloquium, Technische Universität Ilmenau, 08. – 12. September (2014); URN: urn:nbn:de:gbv:ilm1-2014iwk:3
5. H. Lorenz, E. Beckert, R. Schödel: *Phase topography-based characterization of thermal effects on materials and joining techniques*. Applied Optics, accepted (2015)