HIGH TEMPERATURE MATERIAL CHARCTERIZATION BY DYNAMIC MECHANIC ANALYSIS

H. Mucha[1*], H.Deckmann[1]
[1] Netzsch Gerätebau, Ahlden Germany,
* Corresponding author (Herbert.mucha@netzsch.com)

Keywords: *Dynamic mechanic analysis, high temperatures, high forces, test atmosphere*

Abstract

A Dynamic Mechanic Thermal Analyzer of Type EPLEXOR 500-HT (producer: Netzsch, Germany) is used to perform thermal analyses between room temperature and up to 1500 °C. The performance and handling allow quick analyses of the test materials. Here the analyses are restricted on some well known reference materials like Quartz wafer, Sapphire single crystal, polycrystalline Alumina and Inconel 600. The materials are ideally sized about 2 mm x 8 mm x 45 mm. Depending on the test material the maximum temperature varies up to maximally 1500°C. The temperature dependence of the elastic |E*|-Modulus and the damping tanδ of the oxidation resistant materials are determined in ambient air.

1. Introduction

The knowledge of dynamic mechanical material data at elevated application temperatures and under different load amplitudes is requested by different industrial sectors. Among such technical components could be lightweight components, high temperature shields and structural components (i.e. Metal Matrix Composites (MMC), Ceramic Matric Composites (CMC) and monolithic ceramics). All of them need a mechanically characterization for their successful high temperature applications.

The method of Dynamic Mechanic Analysis (DMA) applies a periodic mechanical excitation (deformation) to the test material over the entire temperature range from -160 °C up to maximally +1500°C. It records the periodic response with respect to the mechanical excitation and derives the elastic Modulus |E*| and damping tan δ from the time delay between the amplitudes of excitation and response. Non oxidizing materials are tested in ambient air, whereas oxidizing materials would need an atmosphere that possesses a reduced oxygen concentration to scale down the oxidation.

The large temperature differences which occur during the HT measurements lead to thermal expansion differences between sample and holder. These length differences are not negligible and must be balanced continuously during the test.
Without counter measures, sliding processes between sample and support occur and affect the DMA results. A flexible cardanic bending sample holder is used in order to reduce the sliding processes.
The following DMA investigations are performed with this new flexible cardanic bending sample holder.

2. Equipment

2.1 High Force/ Temperature Instrumentation

The EPLEXOR 500 N High Temperature Dynamic Mechanic Analyzer (DMA) from NETZSCH Analyzing and Testing is equipped with a furnace, operating in air up to 1500°C (Fig. 1,2). The flexible Bending Support is displayed in Fig. 3 together with a cardanic piston arrangement.

2.2 Principle of Operation EPLEXOR®

The DMA EPLEXOR® applies a periodic (sinusoidal) mechanical excitation (e.g. force controlled) to the test material and records the cyclic response (e.g. deformation amplitude) with respect to the mechanical excitation.
In order to avoid breakage of brittle samples, deformation amplitudes in the range of only a few microns (e.g. 5 µm) can be required.

The elastic Modulus |E*| and damping tanδ is calculated from the time delay, the excitation amplitude and the amplitude of the response. In case of small sinusoidal samples excitations, the sample response will be within the linear viscoelastic domain.

Fig. 1: HT EPEXOR 500N with standard furnace (-160 °C – 500 °C), right and for high temperature a furnace (RT-1500 °C) for alternative operation (left)

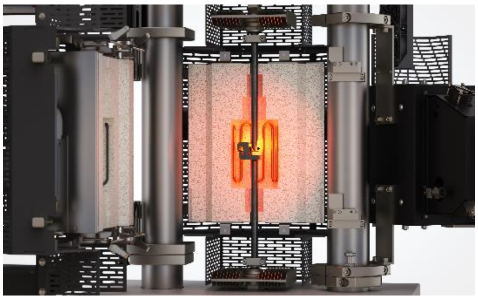

Fig. 2: High temperature furnace (opened), mounted is an asymmetric ceramic bending holder

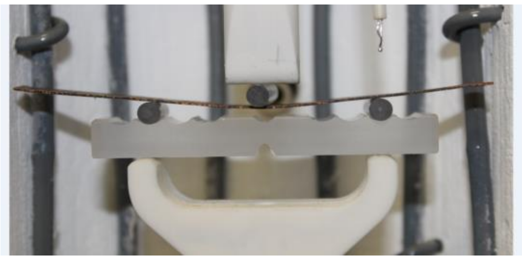

Fig. 3: Flexible Bending Support with Cardanic Piston inside the high temperature furnace

For larger load the material response is not sine shape anymore but it is still periodic. These material answers are evaluated by the EPLEXOR data analysis and determine the modulus E* and damping tanδ.

Unique features of the EPLEXOR® system are the two mechanical drives (servo motor drive for static and an electrodynamic shaker system for dynamic sample loading), which can be controlled independently from each other (Fig.4).

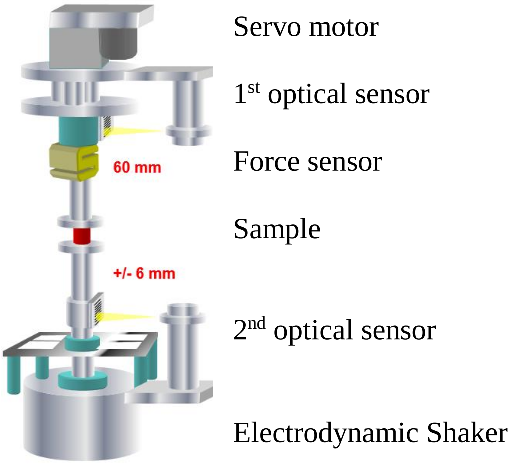

Fig. 4: Principle of Operation of EPLEXOR systems.

Two optical deformation sensors are in use to determine static deformations up to 60 mm and dynamic amplitudes up to +/-6 mm. This assures a wide operation range, high reliability and an excellent accuracy. Interchangeable force sensors with different nominal force ranges make a quick realization of the experimentally best suited test up possible.

3. Results

Different well known materials are investigated by the EPLEXOR®. To indicate the versatility of the HT-DMA specially at high temperatures, a Quartz Wafer, a Sapphire single crystal, a polycrystalline Alumina sample and an Inconel 600 specimen are tested. The results of these analyses are outlined from Fig. 5 to 8.

3.1 Quartz Wafer

Quartz Wafer are fragile and have different thermal expansions along their crystal axes. Therefore Quartz wafer are prone to fracture due to accumulating inner stresses in particular when temperature gradients exist. The EPLEXOR test conditions can be set appropriate to the Quartz test requirements. The Quartz phase transition at 573 °C can be resolved in detail and passed without wafer breakage. The phase transition at 573 °C is sharp and takes place between the trigonal- (α-Quartz) at lower and a hexagonal (β-Quartz) crystal structure at higher temperature [1]. The EPLEXOR HT is equipped for this investigation with a force sensor of 25 N nominal load and passes through this α/β transformation (Fig.5) without breakage.

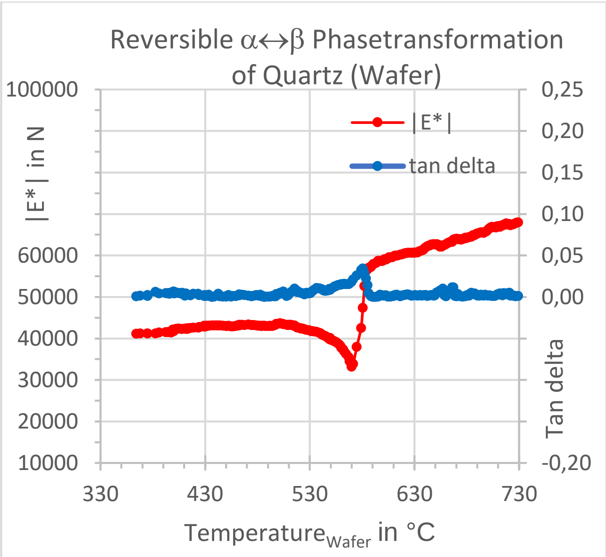

Fig. 5: Reversible α↔ β transformation of Quartz (573°C), 1°C/min

Test conditions: Quartz Wafer
Sample size (wafer) mm: 1,08 x 10,81 x 35,00 3-
3-Point Bending Holder:
Sapphire support with rolls
Span: 3PB-Free 20 mm
Atmosphere: ambient
Max. Temperature: 730 °C
Heating rate: 1 °C/min
Load conditions: $F_{contact}$ = 0.25 N
F_{static} = 0.25 N
$F_{dynamic}$ = +/- 0.15N
Frequency: 5 Hz
Force Sensor: 25 N (nominal load)

During the dynamic mechanic test, the wafer must be handled with care and is not allowed to exposed it to temperature gradients. The homogeneous temperature distribution in the test position in the furnace prevents large temperature gradients and subsequently an internal stress accumulation inside the sample [1].

3.2 Sapphire Single Crystal

The flexible bending support and the test sample are made of single crystalline Sapphire. Sapphire is oxidation resistant.
The expected linear stiffness [2] decrease with increasing temperature without any phase transition proves true (Fig. 6). Therefore, crystalline sapphires are excellent reference materials for recheck the calibration of high temperature DMA systems. Sapphire does not exhibit any phase transition within the entire corresponding temperature range from RT to 1500°C.

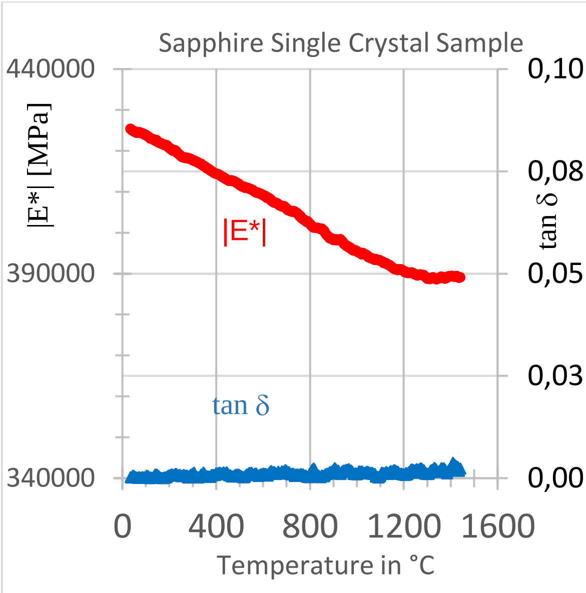

Fig. 6: Temperature dependence of |E*[i]| from a Sapphire Single Crystal sample up to 1450 °C

Test conditions: Sapphire Single Crystal
Sample size: 1 mm x 7,6 mm x 50 mm
3-Point Bending Holder:
Sapphire support and rolls
Span: 40 mm
Atmosphere: ambient
Max. Temperature: 1450 °C
Heating rate: 5 °C/min
Load conditions: $F_{contact}$ = 6 N
F_{static} = 16 N
$F_{dynamic}$ = +/- 4 N
Frequency: 10 Hz
Force Sensor: 150 N (nominal load)
Supplier Info [3]: $|E^*_{RT}|$= 435 000 MPa
Supplier: Saint Gobain Crystals, Milford, NH, USA

3.3 Polycrystalline Alumina

The crystallites which form polycrystalline Alumina, are surrounded by a glassy phase binding them together. The glassy phase softens at elevated temperatures and reduces the stiffness of Alumina. Many attempts are made by the Alumina-makers to reduce the decay in stiffness. The thickness reduction of the glassy layer around the crystallites and filling the glassy phase with fine particles with a high aspect ratio are tried successfully and restrict the sliding of the crystallites [4].

Fig. 7 provides the softening temperature of the tested Alumina type and the corresponding decline of the |E*|-Modulus together with an increase of damping tanδ.

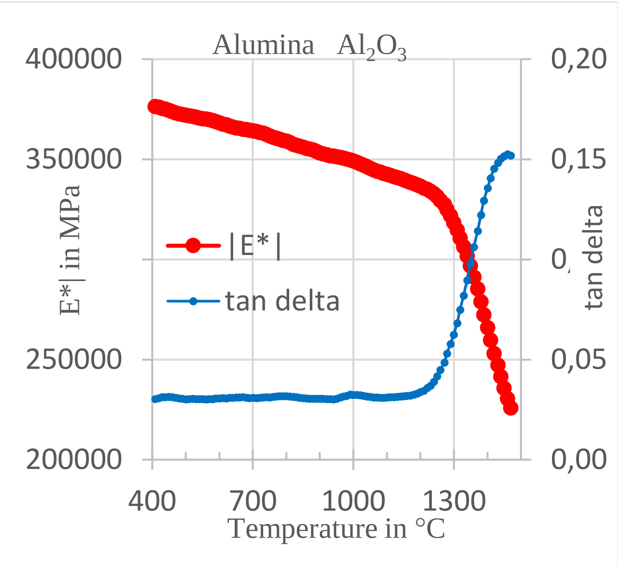

Fig. 7: Temperature dependence of |E*| and tan δ of a polycrystalline Alumina sample up to 1500 °C

Test conditions: Polycrystalline Alumina
Sample size: 3mm x 1,5 mm x 45mm
3-Point Bending Holder:
Sapphire support and rolls
Span: 3PB-Free 30 mm
Atmosphere: Air
Max. Temperature: 1500 °C
Heating rate: 10 °C/min
Load conditions: $F_{contact}$ = 0,3 N
F_{static} = 4 N
$F_{dynamic}$ = +/-2 N
Frequency: 3 Hz
Force Sensor: 25 N (nominal load)

3.4 INCONEL 600

INCONEL 600 is a Ni-Cr-Fe-Alloy. INCONEL is designated for an operation in chemical and thermal severe environments without extreme mechanical requirements.

Fig. 8 shows a slow and continuously, declining modulus E* with increasing temperature for INCONEL600. Up to about 650 °C the damping is also low and constant. The mechanical, thermal behavior of a mechanical part for thermal application therefore is mechanically predictable. The persistence of the corrosion resistance is a priori assured.

In industry Inconel 600 is used preferentially in the temperature range of up to 650 °C.

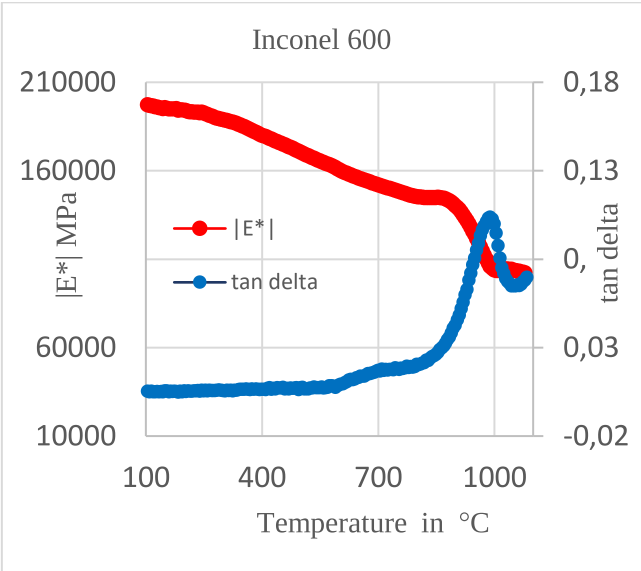

Fig. 8. Temperature dependence of the Modulus |E*| and the damping tan δ from Inconel 600

Test conditions: Inconel 600
Sample size: 8.02mm x 0.96mm x 55mm
3-Point Bending Holder:
Sapphire support and rolls
Span: 3PB-Free 20 mm
Atmosphere: Air
Max. Temperature: 1100 °C
Heating rate: 5° C/min
Load conditions: $F_{contact}$ = 4 N
F_{static} = 4 N
$F_{dynamic}$ = +/-2 N
Frequency: 5 Hz
Force: 150 N (nominal load)

4. Conclusions

- The NETZSCH HT EPLEXOR operates in air atmosphere up to 1500 °C. An upgrade for analyses in oxygen reduced or other atmospheres is possible.
- A special 3-Point bending Holder is used to compensate thermal misfits that occur between the bending support and the sample continuously. It is extremely advantageous for measurements of small deformations and which extend over large temperature ranges.
- Experimental access to mechanical material properties over large temperature regions (RT to max. 1500°C) is routinelly possible.
- Depending on the material´s need the EPLEXOR can apply load amplitudes to explore the transition from the linear and the non-linear material properties.
- Software tools are available to automatize long lasting material analyses in the experimental practice.

5. References

[1] Steffen Klumbach, Elasticity and viscoelasticity of solid SiO_2 as a function of frequency and temperature, Dissertation Karlsruher Institut für Technologie (KIT), 2015

[2] Sapphire data sheet of Saint Gobain Crystals: https://www.crystals.saintobain.com/products/ synthetic-sapphire-materials
Saint Gobain Crystals, Milford, NH, USA

[3] VDM® Alloy 600/600H; Werkstoffdatenblatt Nr. 4107 Revision 01, July2021; VDM Metals, Werdohl, Germany

[4] H. Salmang, H.Scholze; Keramik, Springer, 2007