International Spring School on Forefront Alloys and Advanced Materials for Extreme Conditions

15 – 17 May 2017

Sardinia, Italy

Introduction of new material in space - Qualifaction testing needs

Michael Scheerer

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Short Overview of AAC

Company Profile

AAC is providing R&D services and testing in the field of (composite) materials for Aerospace and terrestrial applications.

Company Location:

TFZ-Technopol Wiener Neustadt
Viktor Kaplan-Straße 2
A-2700 Wiener Neustadt, Austria

Team:

26 employees, thereof 23 scientific and technical staff members

Share Holders:

AC2T research GmbH (Center of Competence for Tribology), staff members

TFZ - Technopol Wiener Neustadt

Regional network for education (university of applied science), research institutions and high-tech companies

Company Profile

Organizational Team Structure:

- **Polymer Composites**
- **Inorganic Composites**
- **Materials- & Components Testhouse**

The excellent qualification and experience of the employees with backgrounds in physics, chemistry, material science, mechanics and electronics, ensures an interdisciplinary approach for working on research and development projects for our customers.

Company Profile

- Material- and Component Testing
- Composite Materials
- Functional Surfaces
- Microstructural Characterisation
- Development of Test Methods and Test Equipment

Our product focus is on material testing and material development for highly specialized niches in Aerospace and terrestrial applications.

Company Profile

- **Mechanical Testing**
- **Space Materials Testhouse**
- **Tribology**
- **Microstructure**

Testing of materials and components in extreme environments is one of our key competences. Specialized tests for all material classes are performed not only for applications in Aerospace but also for terrestrial applications.

↓

External Testhouse for ESA since more than 25 years

Introduction

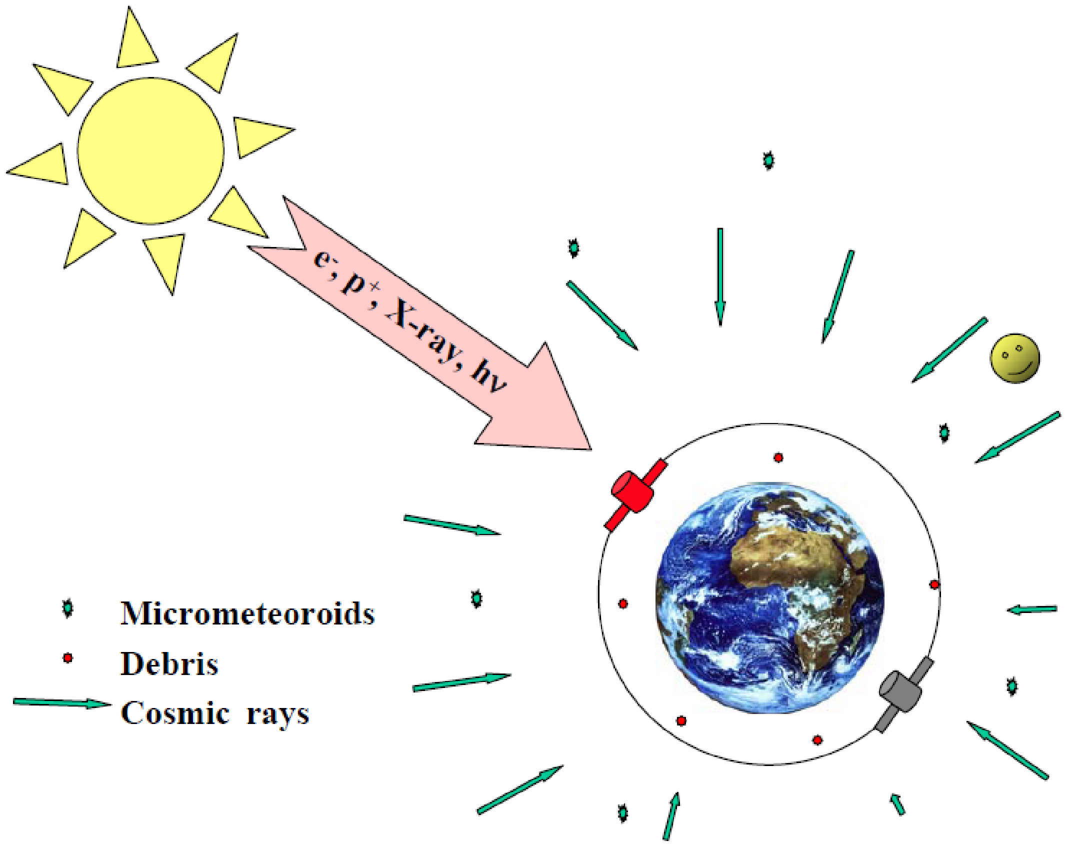
Most materials used in spacecraft are not different from those in terrestrial applications (metallic, ceramics, polymers, composites)

HOWEVER! The operational environment is different! The physical and chemical environment in LEO and GEO is not benign, imposing highly aggressive oxidation and degradation of materials:

- High energy particles and ionizing radiation from the Sun. This hot plasma (or solar wind) is trapped and concentrated in regions of the Earth's magnetic field.
- Thermal waves and gradients exist in the upper atmosphere. For example the International Space Station (ISS) will undergo about 175,000 thermal cycles from +125°C to –125°C as it moves in and out of the Earth's shadow.
- High velocity travelling meteoroids and debris particles form a constant threat to spacecraft.
- Re-entry vehicles for earth and planetary missions may encounter temperatures >1,600°C.
- Often the material application is subject to conflicting requirements (e.g. mass, stability/conductivity, strength, radiation …)
- In many cases each satellite project has to address specific material issues.

Introduction

Space Environment Requirements for Materials in flight



Vacuum

Temperature
(isothermal/cycling)

Radiation

Space Debris
Micrometeoroids
Atomic Oxygen

(Manned
Compartments)

From: Giancarlo Bussu – ESTEC IAASS Quality Assurance Course, November 2008

Introduction

- **Pre-launch and Launch environment**
  - Corrosion & Susceptibility to Stress Corrosion
- **Space Environment**
  - Outgassing of Materials due to Vacuum environment
    - Gas cloud lead to discharge or arcing
    - Deposited material can influence operation (e.g. solar sails, mirrors, thermo-optical properties, …)
    - Parts could shrink due to the release of water (→ Coefficient of moisture expansion - CME)
  - Solar EUV
  - Extreme Temperature
    - Embrittlement at low temperatures
    - Fatigue damage due to thermal cycling
  - Friction & Wear
    - Especially for metals
    - no reformation of oxide layers could lead to sticking in mechanisms (→ cold welding)
  - Impact of micrometeorids or debris
  - Presence of radiation
    - Degradation of material properties

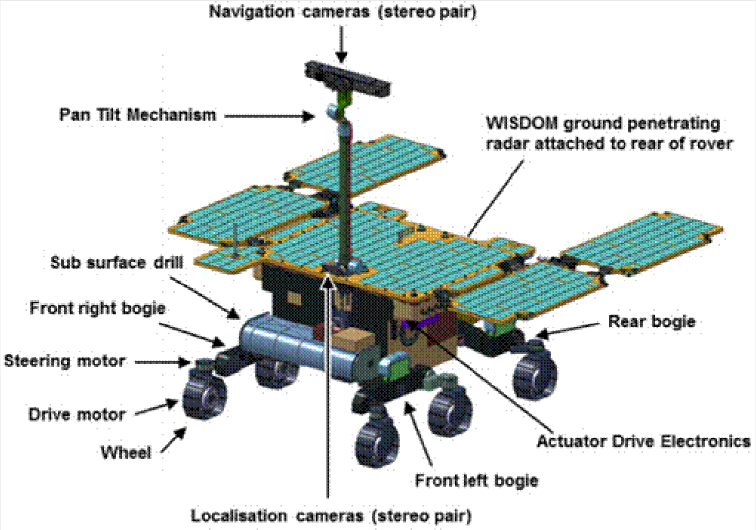
Introduction

ECSS-Q-70B Materials, mechanical parts and processes **ECSS-Q-70-01A** Contamination and cleanliness control **ECSS-Q-70-02A** Thermal vacuum outgassing test for the screening of space materials **ECSS-Q-70-03A** Black-anodizing of metals with inorganic dyes **ECSS-Q-70-04A** Thermal cycling test for the screening of space materials and processes **ECSS-Q-70-05A** Detection of organic contamination of surfaces by infrared spectroscopy **PSS-01-706** The Particle and Ultraviolet (UV) Radiation Testing of Space Materials **ECSS-Q-70-07A** Verification and approval of automatic machine wave soldering **ECSS-Q-70-08A** Manual soldering of high-reliability electrical connections **ECSS-Q-70-09A** Measurement of thermo-optical properties of thermal control materials **ECSS-Q-70-10A** Qualification of printed circuit boards **ECSS-Q-70-11A** Procurement of printed circuit boards **ECSS-Q-70-13A** Measurement of the peel and pull-off strength of coatings and finishes using pressure E-sensitive tapes **ECSS-Q-70-18A** Preparation, assembly and mounting of RF coaxial cables **ECSS-Q-70-20A** Determination of the susceptibility of silver plated copper wire and cable to “red plague” corrosion	**ECSS-Q-70-21A** Flammability testing for the screening of space materials **ECSS-Q-70-22A** The control of limited shelf-life materials **ECSS-Q-70-25A** The application of the black coating Aeroglaze Z306 **ECSS-Q-70-26A** Crimping of high-reliability electrical connections **ECSS-Q-70-28A** The repair and modification of printed circuit board assemblies for space use **ECSS-Q-70-29A** The determination of offgassing products from materials and assembled articles to be used in a manned space vehicle crew compartment **ECSS-Q-70-30A** The wire wrapping of high-reliability electrical connections **ECSS-Q-70-33A** The application of the thermal control coating PSG 120 FD **ECSS-Q-70-34A** The application of the black electrically conductive coating Aeroglaze H322 **ECSS-Q-70-35A** The application of the black electrically conductive coating Aeroglaze L300 **ECSS-Q-70-36A** Material selection for controlling stress-corrosion cracking **ECSS-Q-70-37A** Determination of the susceptibility of metals to stress-corrosion cracking **ECSS-Q-70-38A** rev.1High-reliability soldering for surface-mount and mixed technology
ECSS-Q-70-45A Standard methods for mechanical testing of metallic materials **ECSS-Q-70-46A** General requirements for threaded fasteners	**PSS-01-748** Requirements for ESA-Approved Skills Training and Certification (Electronic Assembly Techniques) **ECSS-Q-70-71A** rev.1Data for selection of space materials and processes

From: Giancarlo Bussu – ESTEC IAASS Quality Assurance Course, November 2008

Examples – HighSSM (MMC materials for planetary exploration)

Objective: most promising metallic based composites (MMC) with high specific stiffness (> 34 x106 m2/s2) to be used on planetary spacecraft such as for Mars surface missions



Testing General:

Incoming inspection (Microstructure, Hardness, Ultrasonic)
Tensile Testing at RT [L, LT, 45°], - 100°C, + 100°C [L]
SCC acc. to ECSS-Q-70-37C, Galvanic Corrosion, CTE; [-150;- +300]°C

Testing Mars Environment:

Exposures; Thermal Cycling under Vacuum; [-120; +125] °C, 100 cycles
Thermal Cycling under 6 mbar CO_2; [-120; +40] °C, 100 cycles
Cryo-Immersion into liquid nitrogen for 8 days duration
Tensile Testing, Fatigue, Wear , Damping-Vibration

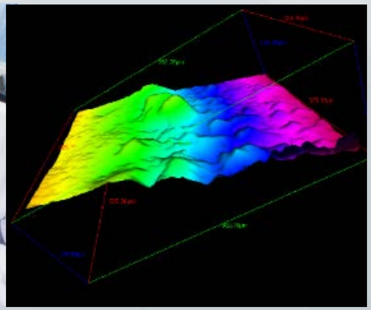
Microstructural Characterisation

- **Materialography**
- **Electron and Ion Microscopy**
- **Surface Analysis**

Our expertise in microstructural characterization is used for material development, failure analysis and quality control. We are working with a huge variety of metals and alloys like steel, Al- and Ti-alloys, composites, coatings and electronic materials allowing evolution of new structural concepts for terrestrial and Aerospace applications.







Optical Microscopy

SEM Microscopy

3-D Surface Analysis

Material & Component Testing
Mechanical Testing

- **Quasistatic Material Testing**
- **Fatigue Testing**
- **Stress Corrosion Testing**
- **Non Destructive Testing**

Mechanical tests for all material classes according to standards are performed. Our specialization is on customer defined testing and testing at extreme temperatures (-269 °C up to 2800°C).
Stress corrosion testing for metallic materials in combination with our microstructural expertise and non destructive testing is another competence.

Quasistatic Testing Testing

Fatigue Testing

Stress Corrosion Test

Non Destructive

Material & Component Testing
Mechanical Testing

Property / Test method	Environment
Tensile testing • Tensile strength • Young´s Modulus	-269 … 1100 °C
Compression tests • Compression strength • Compression Modulus	-269 … 1100 °C
Bending tests • Bending strength • Bending Moulus • 3-pnt and 4-pnt bending	-269 … 1100 °C
Charpy Impact Test	-196 °C / RT
Fracture Mechanic Properties • KIC, KJC, J-Integral • Fatigue crack growth	-269 … 1100 °C
Special Composite Properties • ILSS • Two Rail Shear • TestPicture frame test	-269 … 1100 °C

Climate chamber
-150 °C … 600 °C

LN2 cryostat
-196 °C

LHe cryostat
-269 °C

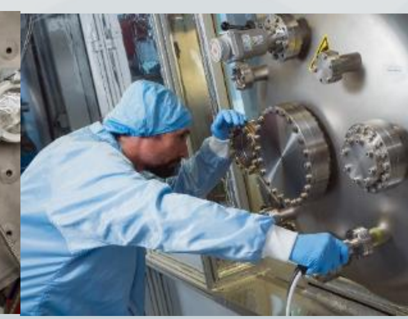
Material & Component Testing
Space Materials Testhouse

- **Outgassing**
- **Thermal Cycling**
- **Extreme Environments**
- **Component Testing**

The experience as Space Material Testhouse for the European Space Agency (ESA) serves as basis for developing customer specific solutions. In our specialized testing facilities we are able to perform tests from material-level up to flight hardware in clean room environment.







Outgassing

Thermo Mechanical Tests

Component Testing

Material & Component Testing
Space Materials Testhouse

Simulation of thermal vacuum conditions e.g. space environment in open space and sun exposure.
To fulfill different requirements, a range of test facilities is available at AAC:

Test rig	Size of heating / cooling plate(s)	Temperature range	Remarks
TVC	2 plates 100x70cm	-180 … +350°C	Attached to clean room class 7
LVC	2 plates 100x1000cm	-180 … +350°C	
Outgassing	30cm diameter	-180 … +300°C	
WRK	50x50cm	-180 … +300°C	Inductive heating available (up to 2500°C) 70kN servo-hydraulic test machine attached
miniWRK	35x30cm	-180 … +500°C	




Re-Entry Simulation Chamber

Simultaneous application of

- Temperatures up to 2,500°C, heating rates up to 500 K/sec
- High vacuum, inert or reactive gases up to 1 bar
- Static or dynamic mechanical loads up to 70kN and 70Hz

Heating

- 30 kW induction heater
- Up to 2,500 °C
- Heating rate up to 500 K/min (depending on test article)
- Direct heating, inside susceptor tube, or via blackbody irradiation
- Temperature control by pyrometer or thermocouples

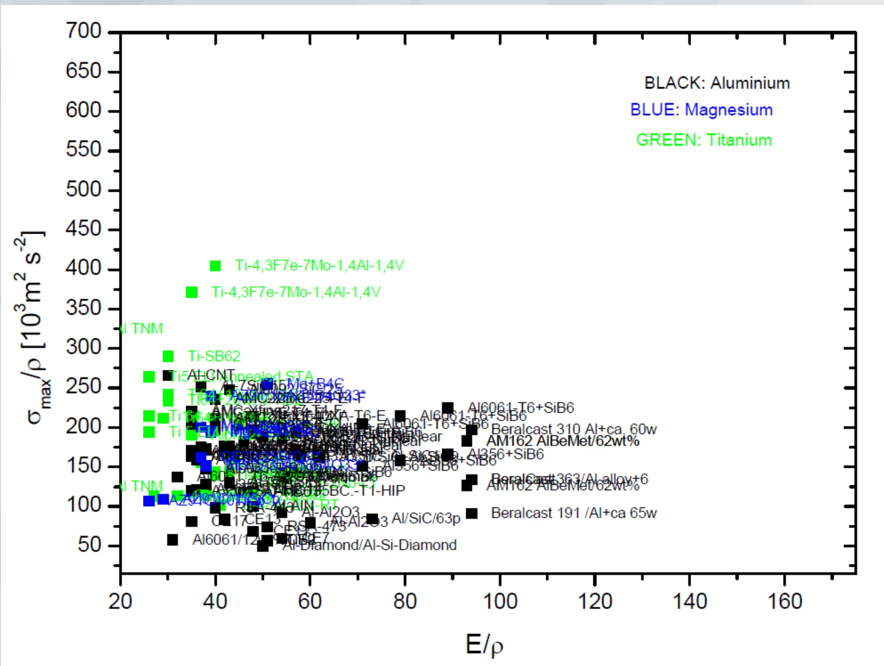
Available atmospheres

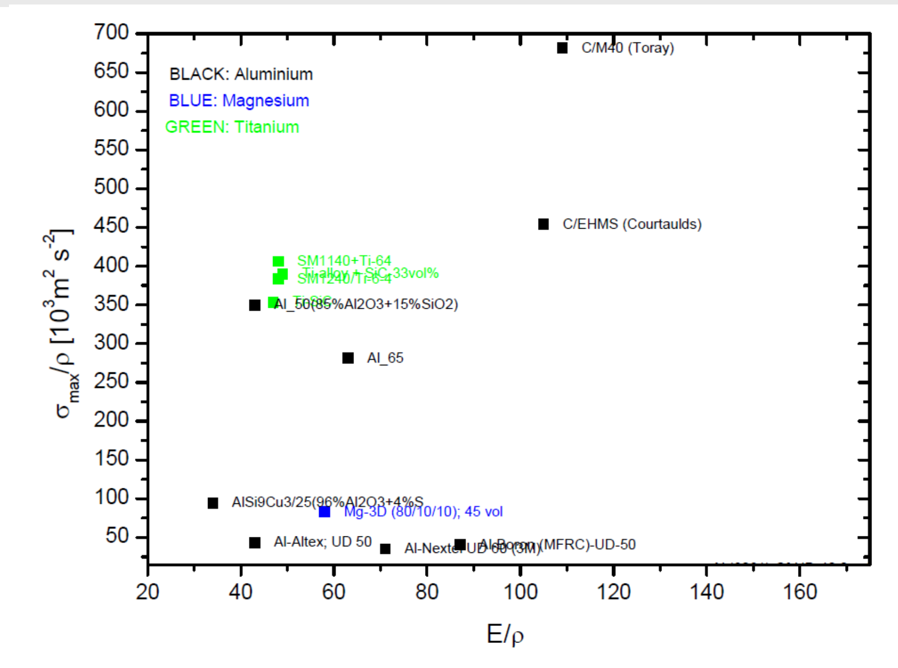
- High vacuum (10–6 mbar)
- Inert gas (e.g. Ar, CO_2, air, …)
- Reactive gases (e.g. Ar/H2 mixtures)
- Automatic pressure and/or flow control

Static or dynamic mechanical loads

- Tensile/compression/shear loads up to 70kN
- Static or dynamic loading, frequency up to 70Hz
- Strain measurement by laser speckle or strain gauge

Examples – HighSSM (MMC materials for planetary exploration)



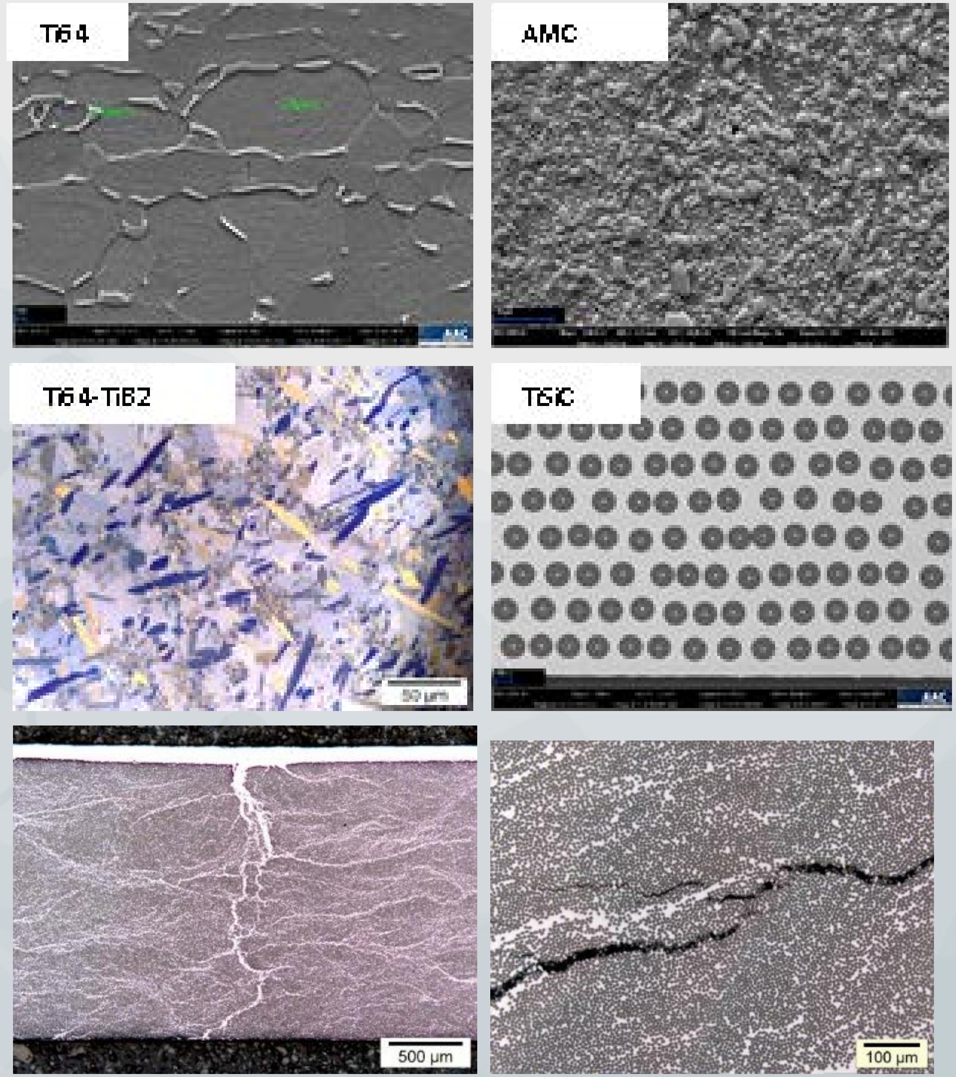


σ_{max} / ρ ("Ashby D") versus Stiffness Parameter E / ρ for all Reviewed HSSM MMC / Materials, Particle vs. Fiber Reinforced HSSMs

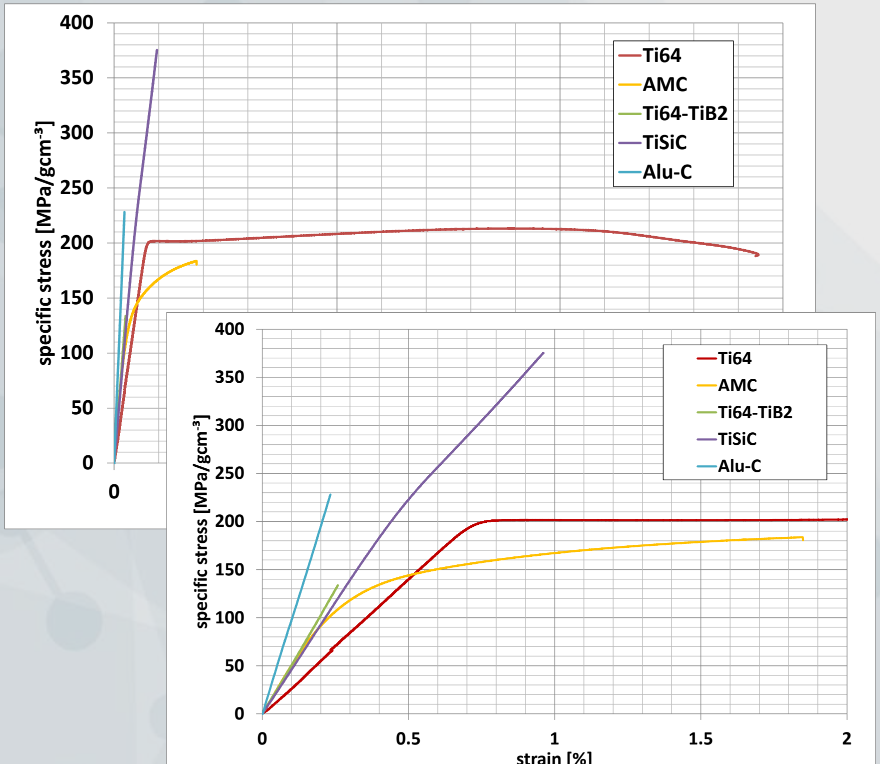
Examples – HighSSM (MMC materials for planetary exploration)

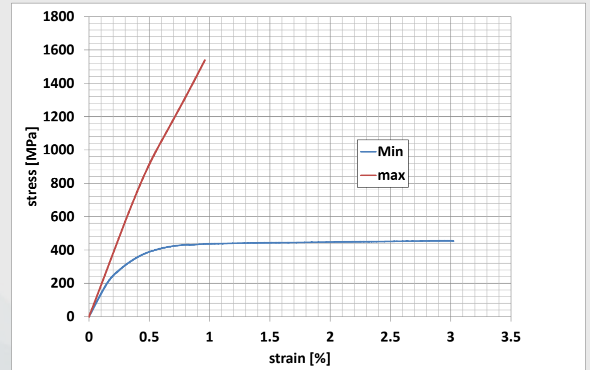
Materials selected for testing earth

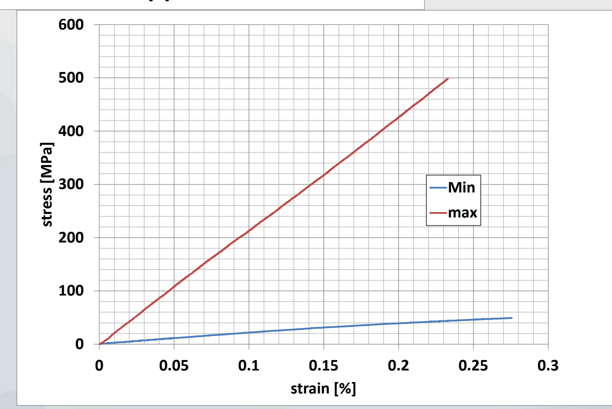
Al-MMCp –
AMC 640xa (AA6061 + 40vol% SiC)
Al-MMCf –
(Al 99,85 + 60vol% C/M40 fibres)
Ti-MMCp –
(Ti6Al4V + 30-35 vol% TiB2)
TiMMCf –
(Ti3Al2.5V + 33-35vol % SiC fibres)



Examples – HighSSM (MMC materials for planetary exploration)



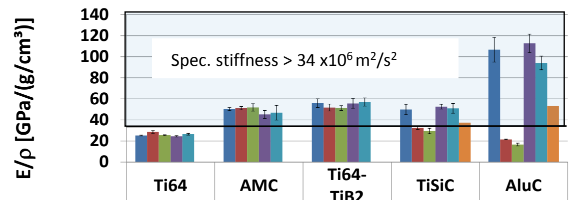




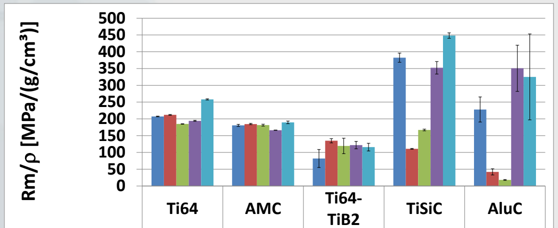
Ti64: expected behaviour / low specific stifffness
AMC: expected behaviour (highest strain of MMC)
Ti64-TiB2: etremely brittle (high spec. stiffness)
TiSiC: highest strenght
Alu-C: brittle / highest spec. Stiffness /strenght much lower than expected (2000 – 2500 MPa)

Examples – HighSSM (MMC materials for planetary exploration)

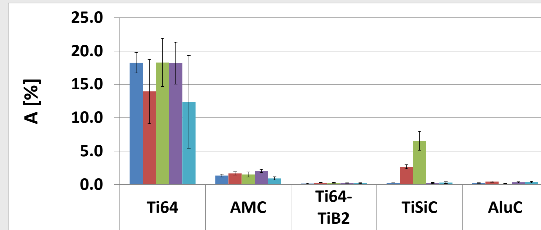
ICARUS SUPERMAT



	Ti64	AMC	Ti64-TiB2	TiSiC	AluC
EL/ρ	25.21	50.27	55.87	49.94	106.67
ET/ρ	28.57	51.18	51.77	32.43	21.49
E45/ρ	25.49	51.87	51.27	29.48	16.66
EL,100°C/ρ	24.45	45.26	55.64	52.63	112.77
EL,-100°C/ρ	26.43	46.89	57.01	50.98	94.15
EL/ρ,quasi				37.45	53.33

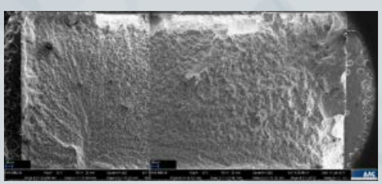


	Ti64	AMC	Ti64-TiB2	TiSiC	AluC
RmL/ρ	207.32	180.58	81.88	382.38	227.95
RmT/ρ	211.92	184.58	134.82	110.69	42.01
Rm45/ρ	184.87	181.46	119.36	166.93	18.11
RmL,100°C/ρ	194.19	166.22	121.84	352.29	350.73
RmL,-100°C/ρ	258.01	189.51	115.96	448.23	325.01

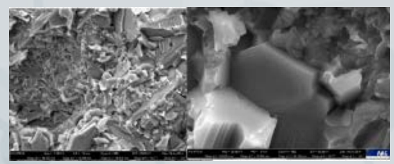


	Ti64	AMC	Ti64-TiB2	TiSiC	AluC
AL	18.25	1.33	0.14	0.23	0.22
AT	13.95	1.66	0.26	2.65	0.43
A45	18.27	1.49	0.23	6.52	0.11
AL,100°C	18.19	2.01	0.22	0.23	0.32
AL,-100°C	12.38	0.91	0.21	0.28	0.34

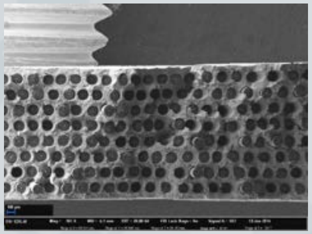
AMC



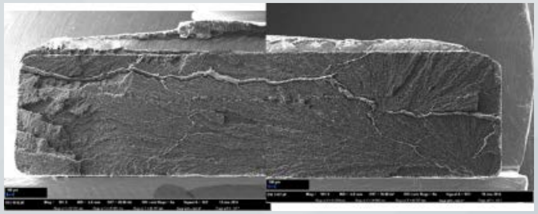
Ti64-TiB2



TiSiC



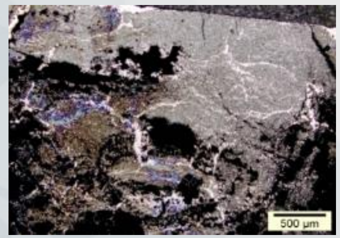
Alu-Cf



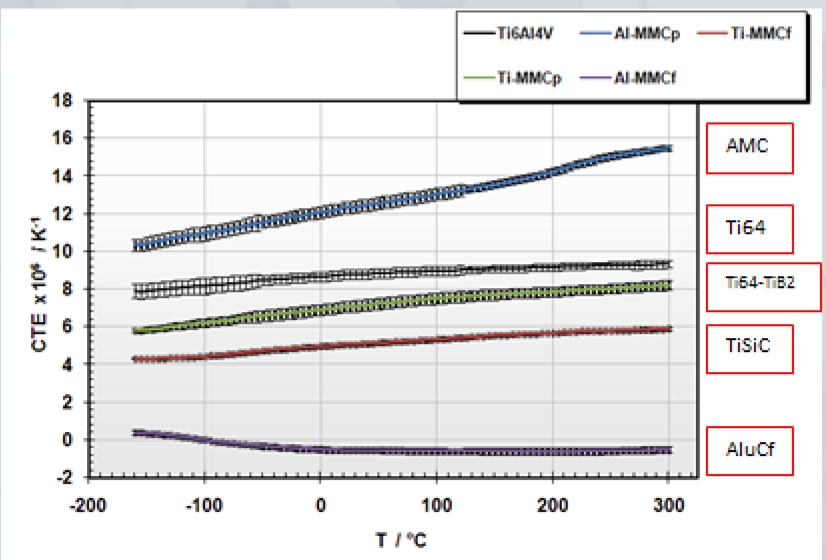
AluCf: highest spec. stiffness, highest anisotropy, spec. strength below the expectation
Ti64-TiB2: sufficient spec. stiffness, low anisotropy, low spec. strength, very low A value,
AMC: sufficient spec. stiffness, low anisotropy, medium spec. strength,
TiSiC: sufficient spec. stiffness, high spec. strength (L), anisotropy- better then AluCf, highest A value

Examples – HighSSM (MMC materials for planetary exploration)

Material	SCC - class	Comments
Ti64	A	ECSS-Q-ST-70-36C
AMC	A	AAC former projects
Ti64-TiB2	A	very brittle, 2 SCC specimens broken during load applying, 1 specimen survived , no tensile test
TISICS	A	neither corrosion nor SCC
AluC	A	severe corrosion of Al, very high amount of corrosion products on the surface



TiSiC exhibits the best performance of all three composites tested . AluCf shows severe corrosion of Al-matrix. Ti64-TiB2 – too brittle for testing

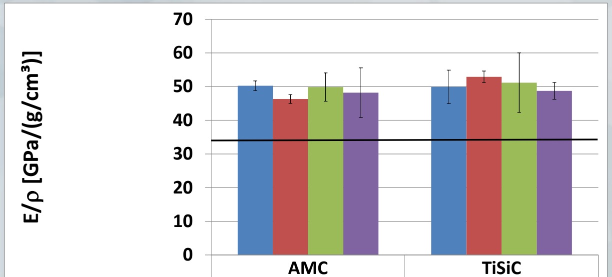


Testing Earth		
Material	CTE	
	-150 and at +300°C x106/T	Comments
Ti64 Ref	7,9 - 9,4	moderate
AMC-SiC (40 vol%)	10,4 - 15,2	high - highest from all materials
Ti64-TiB2 (30-35 vol%)	5,9 - 8,2	moderate
TISICS (42 vol %)	4,3 - 5,9	low
AluC (60 vol%)	0,8 - -0,5	very low / negative

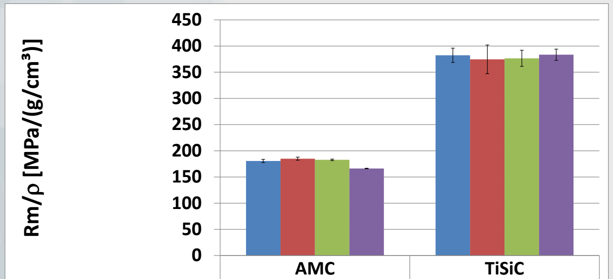
Examples – HighSSM (MMC materials for planetary exploration)

Testing Mars – Results:

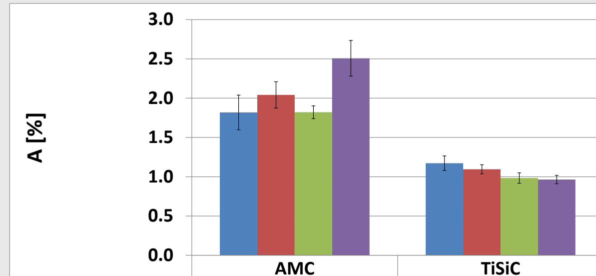
After exposures, no visible degradation of the samples had been observed



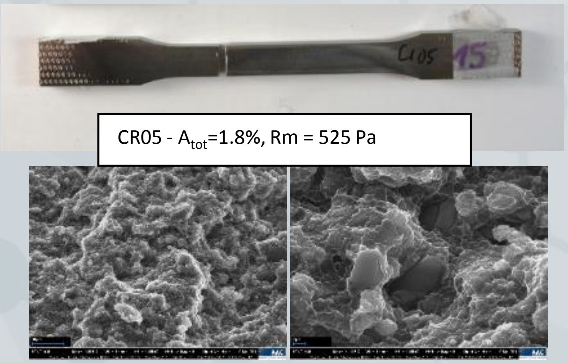
	AMC	TiSiC
EL/ρ as received	50.27	49.94
EL/ρ cryo immersion	46.32	52.90
EL/ρ vacuum cycled	49.87	51.17
EL/ρ CO2 cycled	48.20	48.72

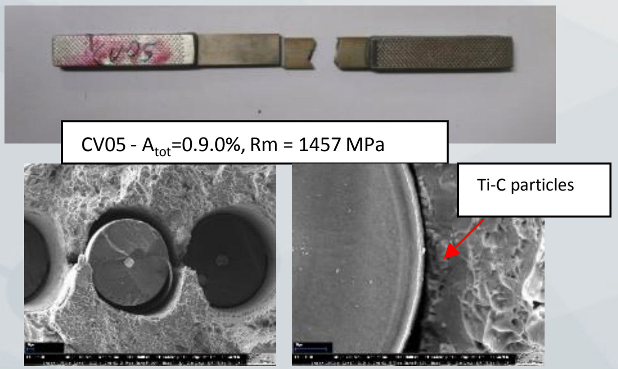


	AMC	TiSiC
Rm/ρ as received	180.58	382.38
Rm/ρ cryo immersion	184.96	374.62
Rm/ρ vacuum cycled	182.74	376.59
Rm/ρ CO2 cycled	166.22	383.40



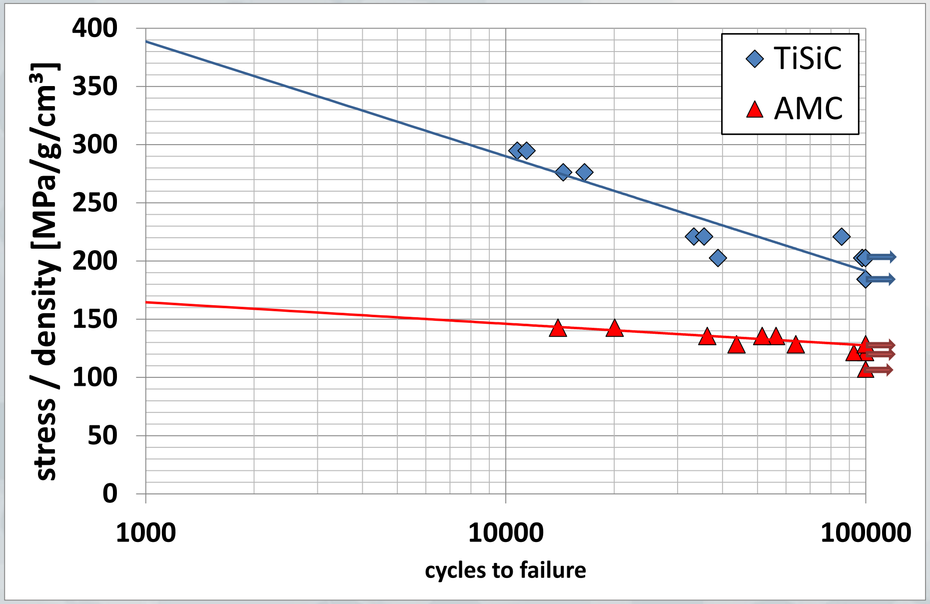
	AMC	TiSiC
A as received	1.82	1.17
A cryo immersion	2.04	1.10
A vacuum cycled	1.82	0.98
A CO2 cycled	2.51	0.96

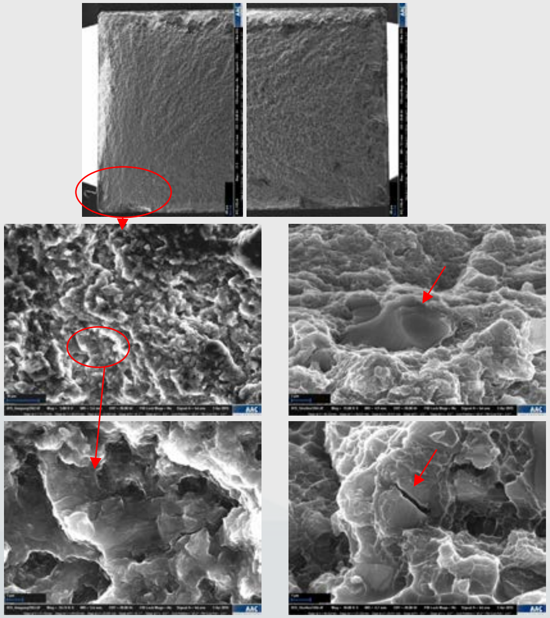


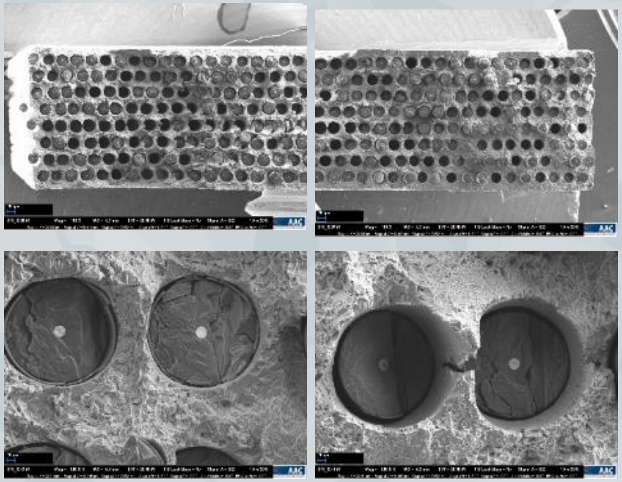


No statistical relevant degradation of the mechanical properties – specific young´s modulus, specific ultimate strength or total fracture strain due to the different thermal treatments; thermal cycling in vacuum, thermal cycling under martial environment or cryogenic immersion in LN2 – has been observed

Examples – HighSSM (MMC materials for planetary exploration)







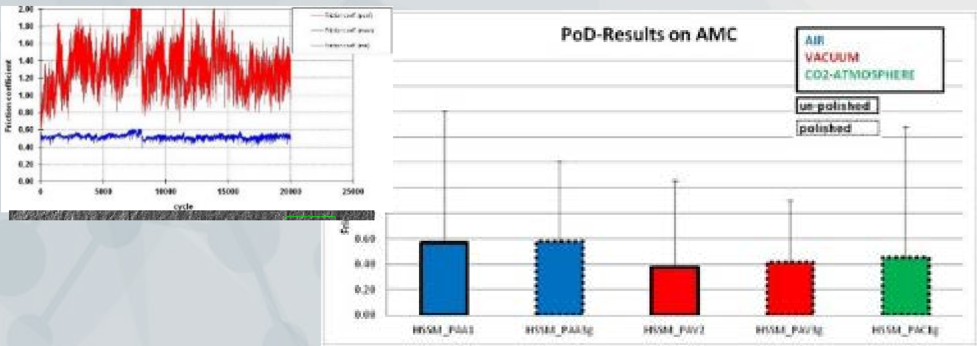
The slope of the specific S-N curve is higher for TiSiC materials indicating a higher sensitivity to fatigue compared to AMC

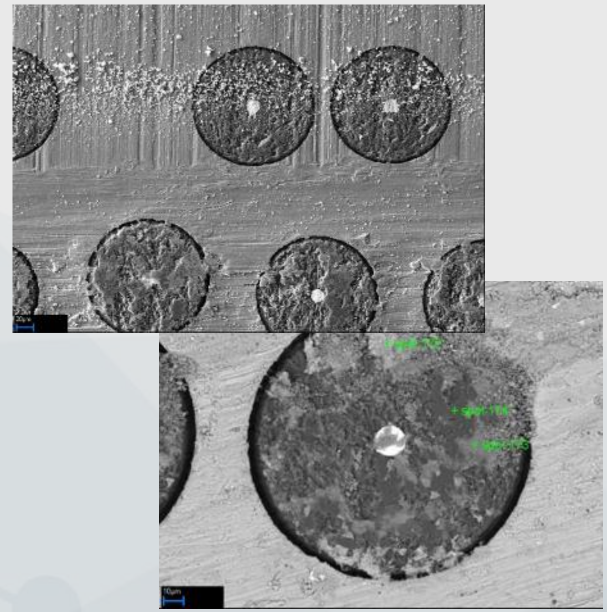
The specific S-N curve for TiSiC shows higher specific fatigue levels in L-direction compared to AMC at the same cycles to failure, which is expected as the specific static strength of TiSiC is more than a factor of 2 higher

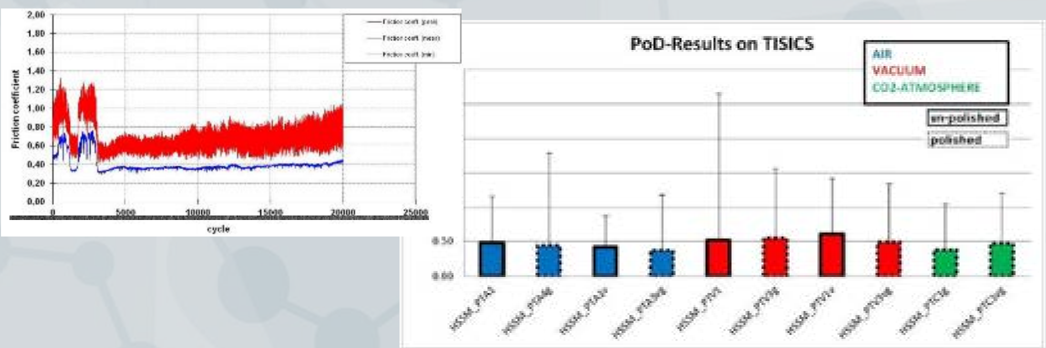
Examples – HighSSM (MMC materials for planetary exploration)

Wear

PoD and Bush tests (against hard steel, uncoated, no lubrication) in air, vacuum and CO_2



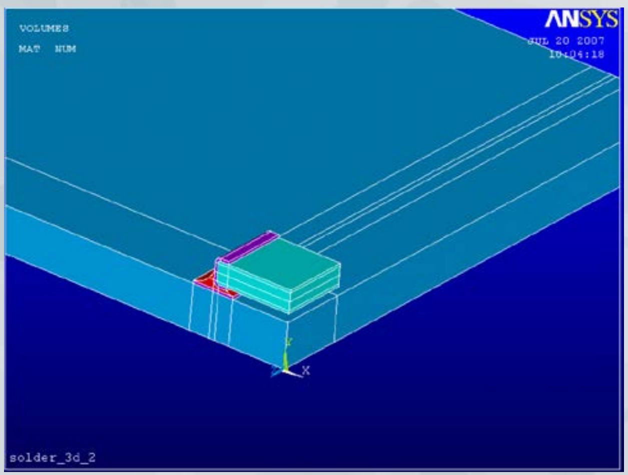


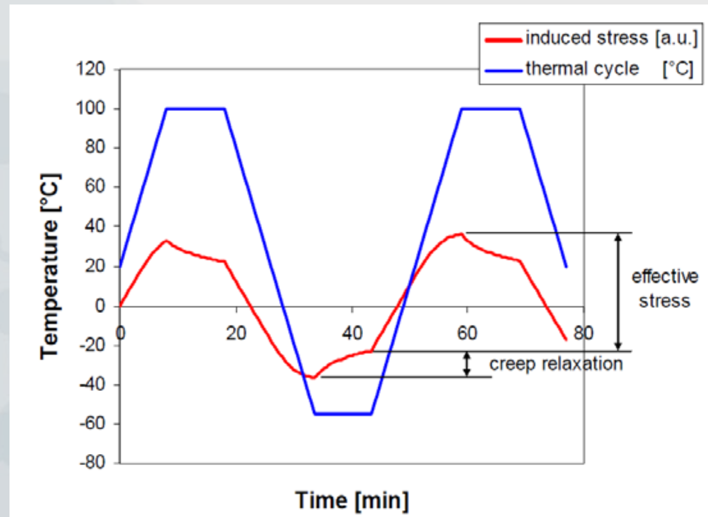


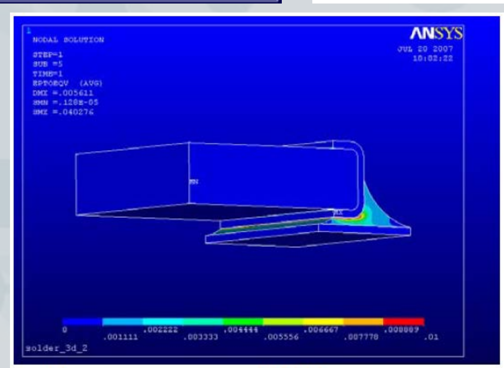
AMC: high wear (particles seem not to reduce wear), high friction (high noise)
TiSiC: fibres reduce wear from Ti to Ti-MMC but high wear on steel ball lower on both after polishing (SiC fibres no more abrasive) low in vaccum and in CO2 when sliding along the fibres high friction (with noise) in all environments

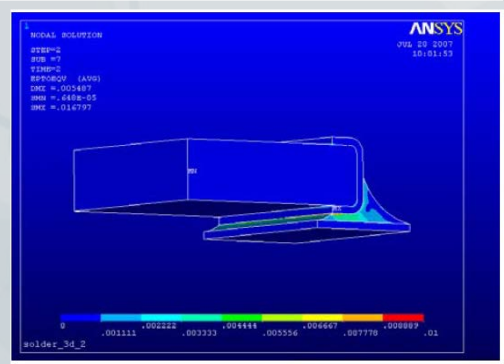
Examples – Testing of electronic material at extreme temperature

Objective: Possibility to extend the operating temperature of conventional and lead free solder materials o be used in space applications





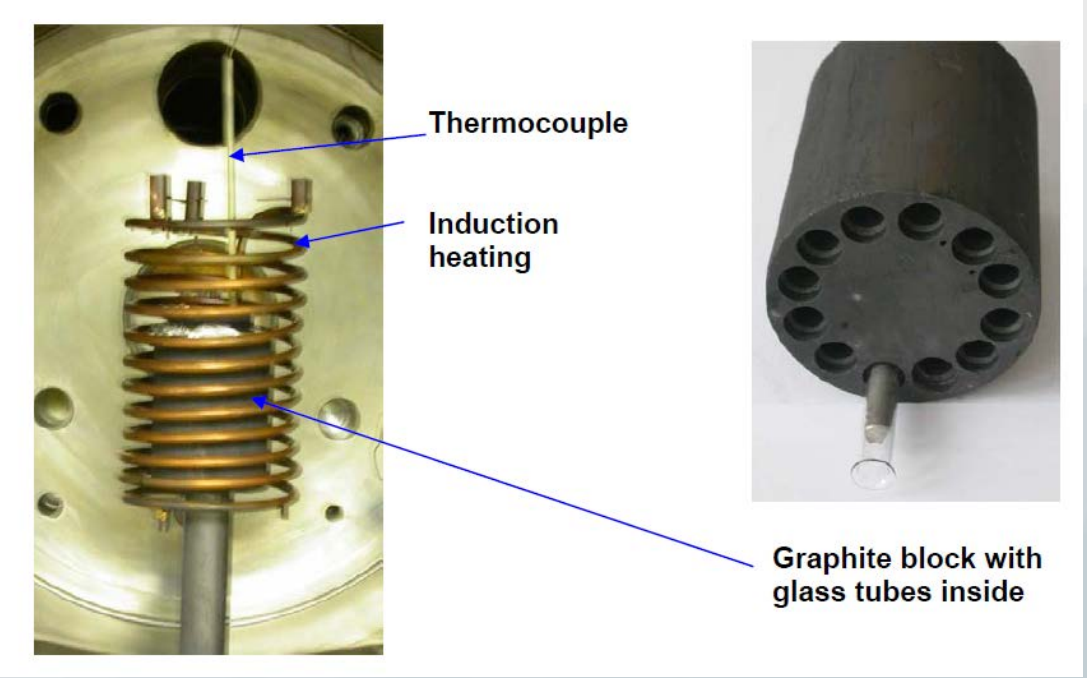




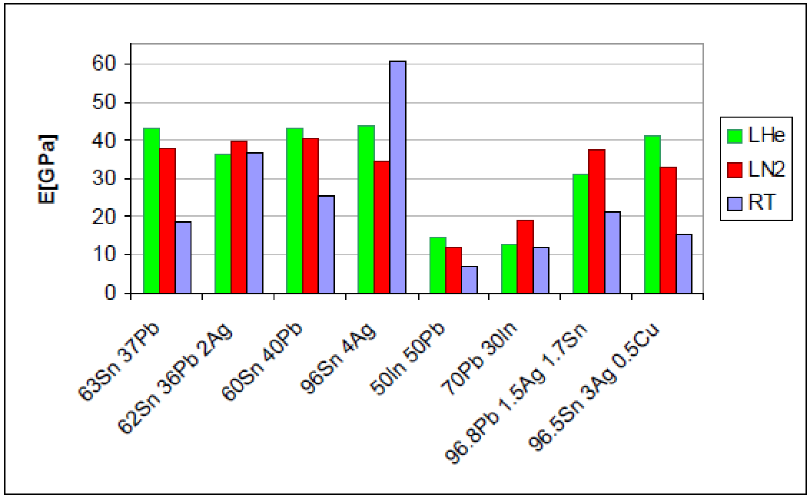
Examples – Testing of electronic material at extreme temperature

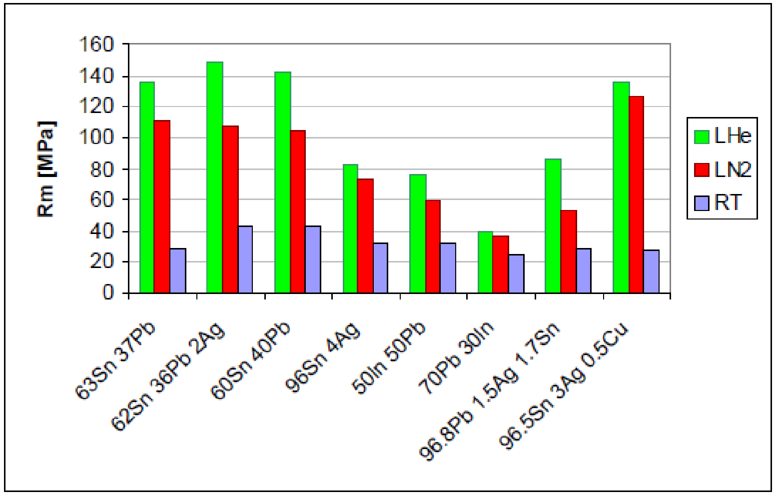
Cryogenic test of electronic materials

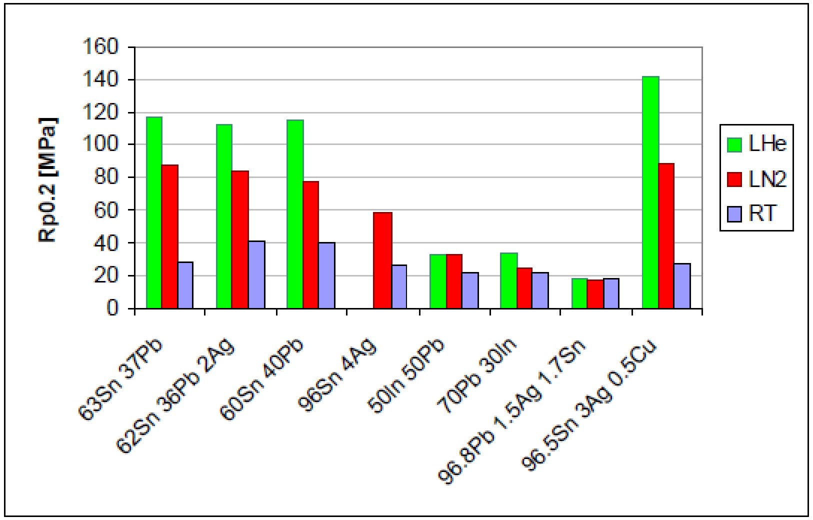
Material		Tensile tests			LO Analysis	SEM Analysis
No.		RT	LN2	LHe		
	Solders [1]					
1	Oxygen-Free High Conduc-(OFHC)copper	3	3	3	x	
2	63Sn 37Pb	3	3	3	x	
3	62Sn36Pb 2Ag	3	3	3	x	
4	60Sn 40Pb	3	3	3	x	
5	96Sn 4Ag	3	3	7	x	
6	50In 50Pb	3	3			
7	70Pb 30In	3	3			
8	96.8Pb1.5Ag1.7Sn	3	3			
19	96.5Sn3.5Ag0.5Cu	3	3			
	PCB [2]					
9	MLB polyimide glass fibre	3	3			
10	MLB epoxy glass fibre	3	3			
11	MLB Thermount	3	3			
	Conformal coatings [3]					
12	Arathane 5750	3	3			
13	Sylgard 184	3	3			
14	Scotchcast 280	3	3			
15	Solithane 113	3	3			
16	CV-1144-0	3	3			
17	Mapsil 213	3	3			
18	Conathane EN4/EN11	3	3			

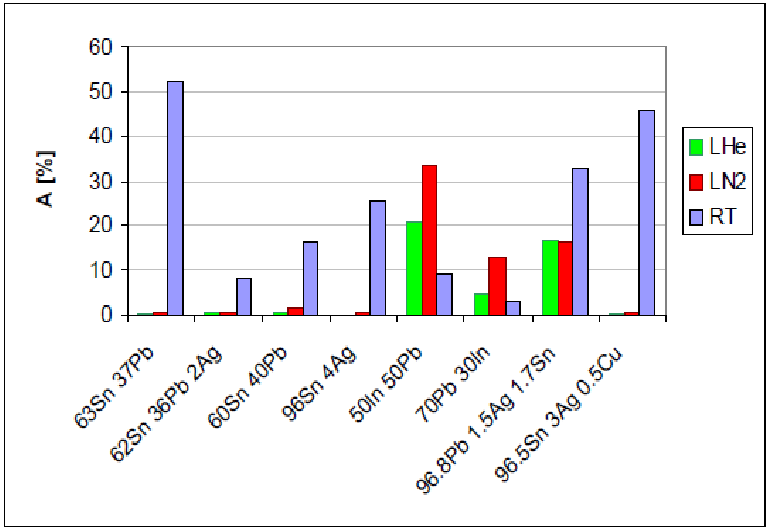


Examples – Testing of electronic material at extreme temperature

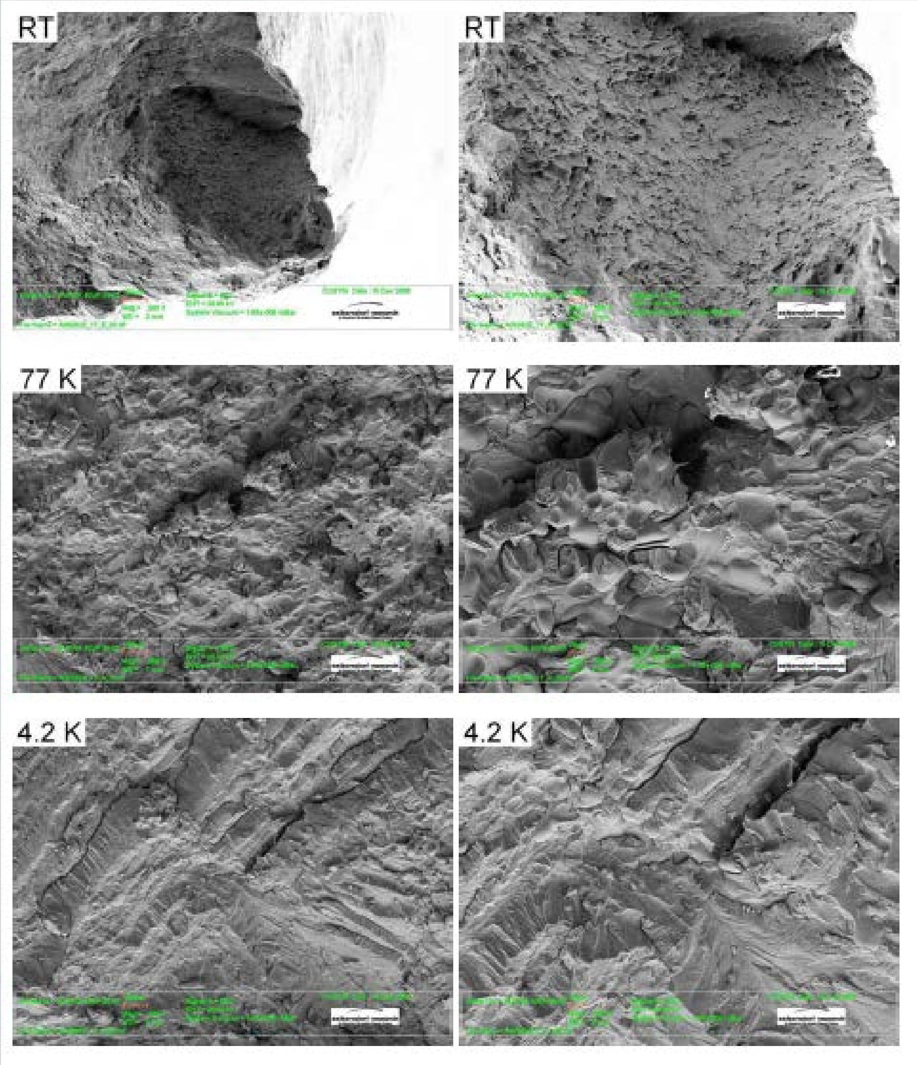








Examples – Testing of electronic material at extreme temperature

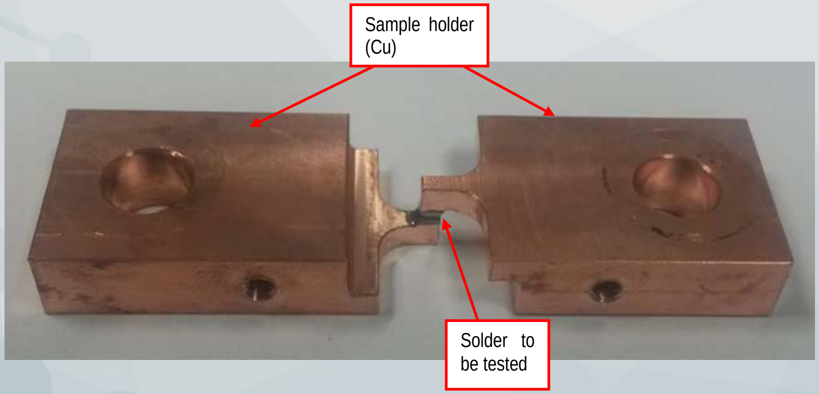


From ductil to very brittle failure

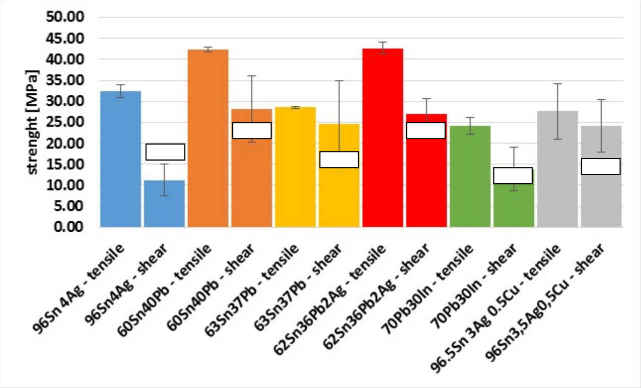
Examples – Testing of electronic material at extreme temperature

Material	TC	Shear test										
		as received	after TC	after Creep @ RT, load 1	after Creep @ RT, load 2	after Creep @ RT, load 3	after Creep @ HT1, load 1	after Creep @ HT1, load 2	after Creep @ HT1, load 3	after Creep @ HT2, load 1	after Creep @ HT2, load 2	after Creep @ HT2, load 3
62Sn36Pb2Ag	TC1	5	5									
63Sn37Pb	TC1	5	5	3	3	3	3	3	3			
60Sn40Pb	TC1	5	5									
Sn96.5Ag3Cu0.5	TC2	5	5									
Pb90Sn10	TC2	5	5	3	3	3				3	3	3
Pb96.8Ag1.5Sn1.7	TC2	5	5									
Pb70In30	TC2	5	5	3	3	3				3	3	3

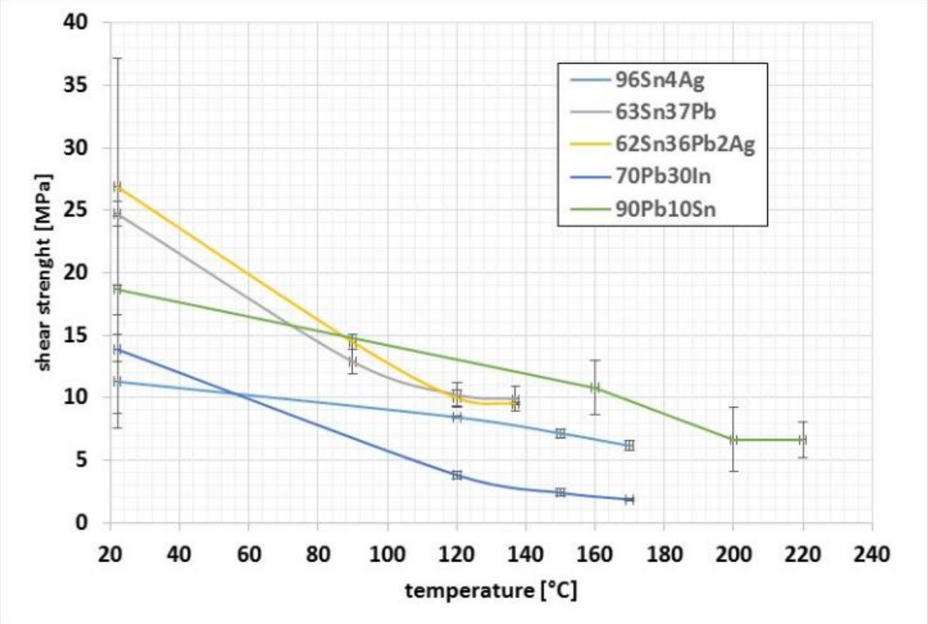
- First group ("normal" solders):
  - Thermal Cycling TC1: -55 °C / +125 °C, 15 min dwell time, max. 10 °C/min ramp rate: 100 cycles
  - Creep @ HT1 – 120°C: 100 d @ 120 °C with 0.5 / 1 / 2.5 N load (values defined acc. to RD08)
- Second group ("high temperature" solders):
  - Thermal Cycling TC2: -55 °C / +150 °C, 15 min dwell time, max. 10 °C/min ramp rate: 100 cycles
  - Creep @ HT2 – 160°C: 100 d @ 160 °C with 0.5 / 1 / 2.5 N load (values defined acc. to RD08)

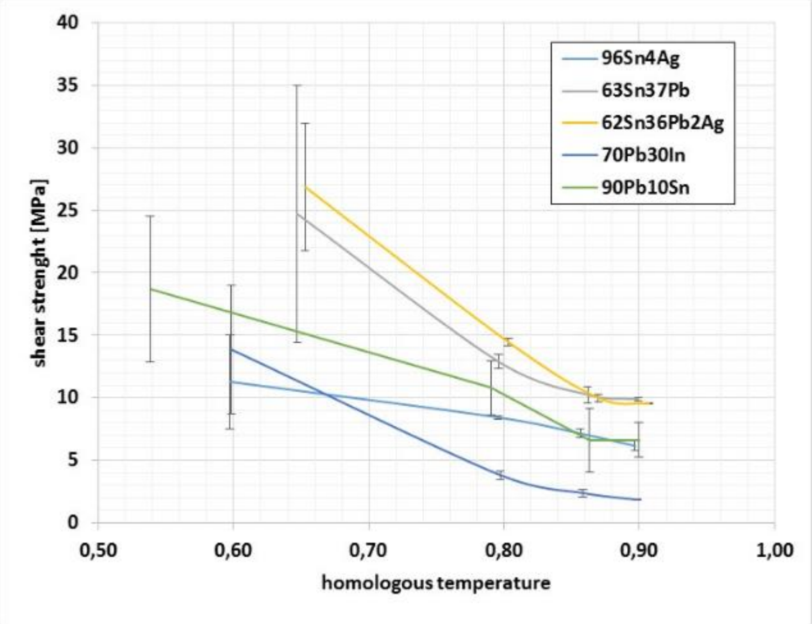


Examples – Testing of electronic material at extreme temperature



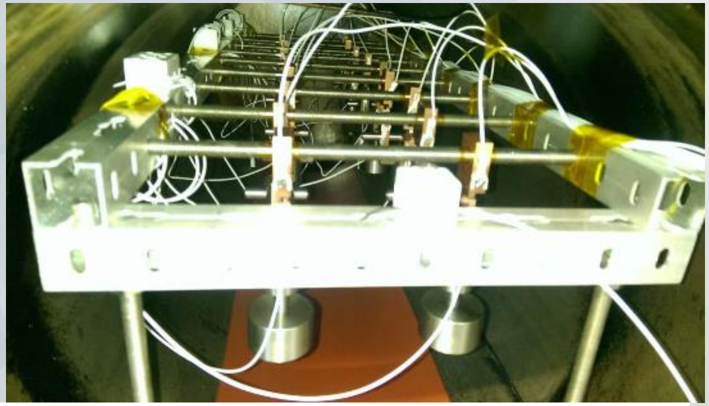
Comparison between tensile strength (CO 55) and shear strength



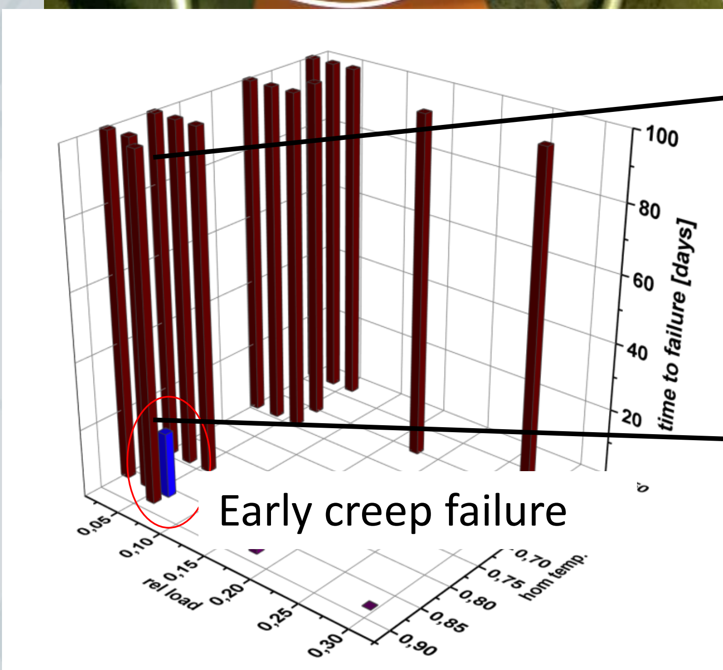


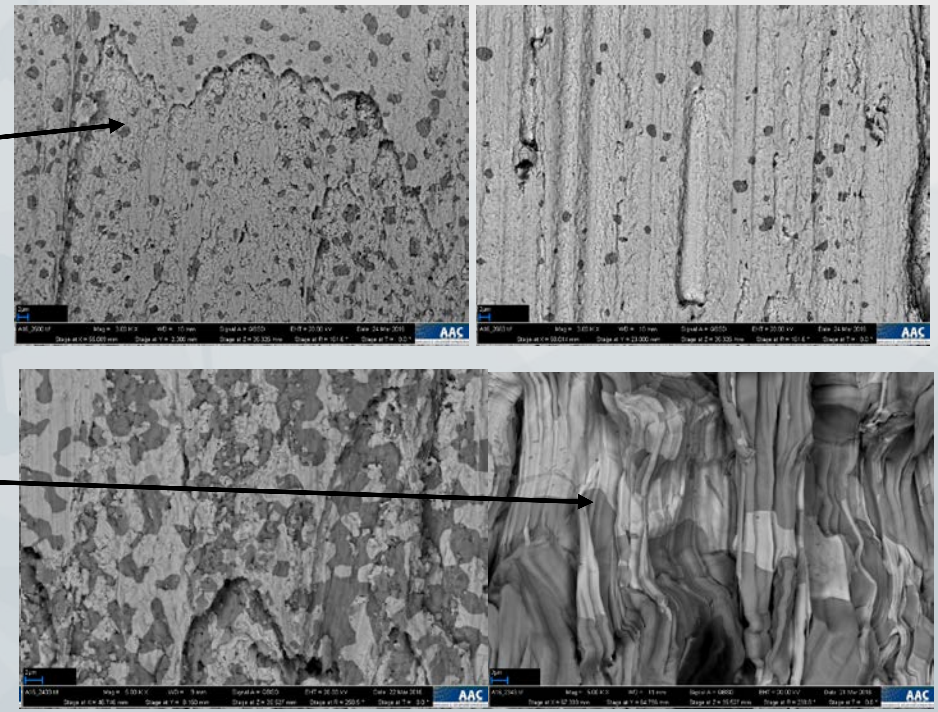
Temperature dependence of shear strength of selected solder materials

Examples – Testing of electronic material at extreme temperature



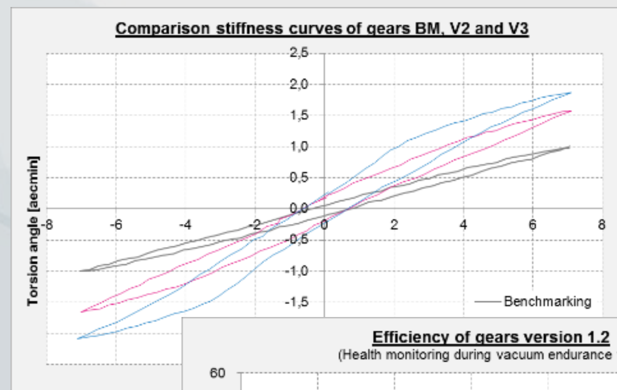
Dependence of creep failure on homologouse temperature and relative shear load – but microstructure (eutectic / non eutectic very important)

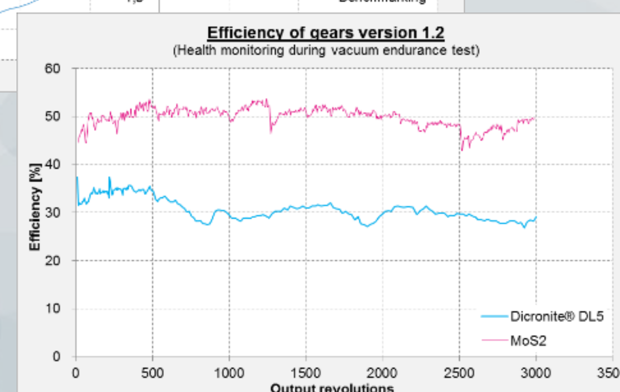




EU-FP7 PROJECT: DRY LUBRICATED HARMONIC DRIVES FOR SPACE APPLICATIONS

Development of solid lubricant coatings for Harmonic Drives in space. Currently, the use of grease lubrication is linked with risk of outgassing, contamination of other parts and limits the usage in temper-atures approx. -50°C to +70°C. The use of solid lubricants typically for bearings may extremely widen the usage to at least -170°C to 300°C.





- **Space Tribology**
- **Electro Tribology**
- **Forming Tribology**

For use in space applications, materials and coatings can be tested from sample level up to semi-components (gears or bearings). Testing of electrical components for space and on-ground applications (sliding contacts, slip rings, brushes…) is another competence in tribology. For optimizing production processes in metal forming, measurement of friction factors during the forming process can be done.






Vacuum Tribometer — Gear Box Test — Electro Tribology — Forming Tribometer

Material & Component Testing
Tribology

Tribology on (dry lubricated) bearings and electrical sliders

- Development of composite materials and coatings
- Characterization of materials intended for bearings
- Applications: Journal bearings, cages for roller bearings, sliding elements, electrical sliders

Tribology in static contacts (subjected to fretting)

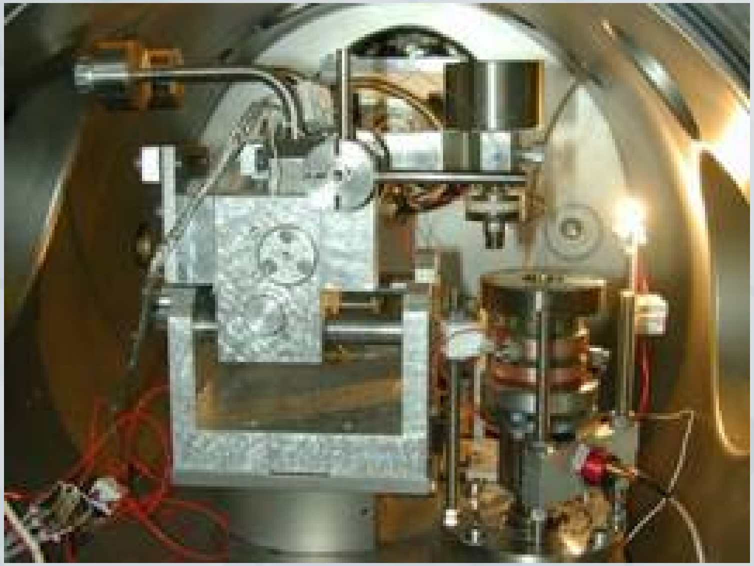
- Characterization of materials intended for “static contacts”
- Development of anti-adhesion and anti-fretting-coatings
- Applications: Hold down points, fretting contacts, cutting

Tribology in forming

- Measurement of friction in metal forming and cutting
- Simulation of Friction in Metal Forming and cutting (Partner)
- Development of tool materials & coatings

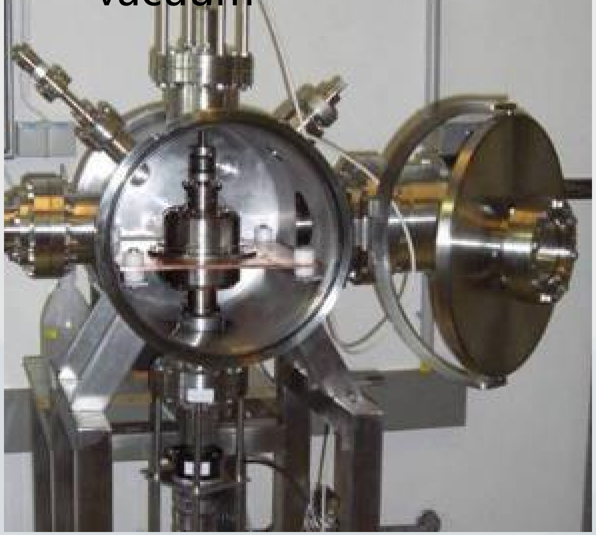
Examples for vacuum-tribological test rigs

Vacuum – Pin-on-disc



Vacuum-Fretting

Gear testing under vacuum



MMC development for space

- **Space Testing**
- **Tribological System Design**

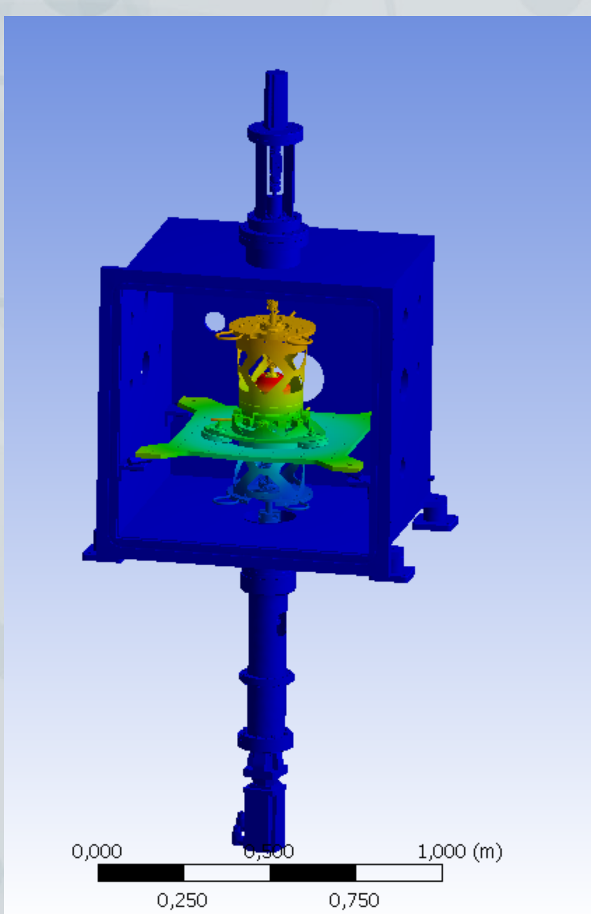
Performing highly specialized tests in extreme environments often requires the development of new test methods.

Together with our clients the requirements are defined, existing test methods adapted or new and innovative measuring procedures and functional tests are developed.

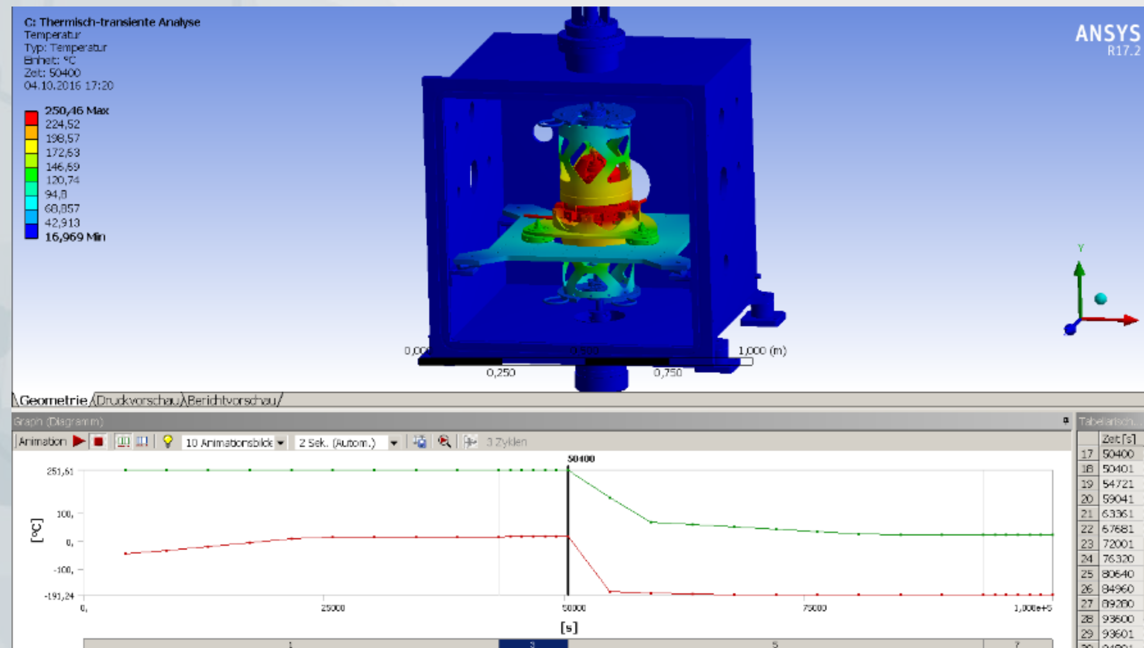
Design and realization of test equipment with our network of specialized suppliers.

Examples – Harmonic Drive Gears for Space

In-house Design of new test facilities

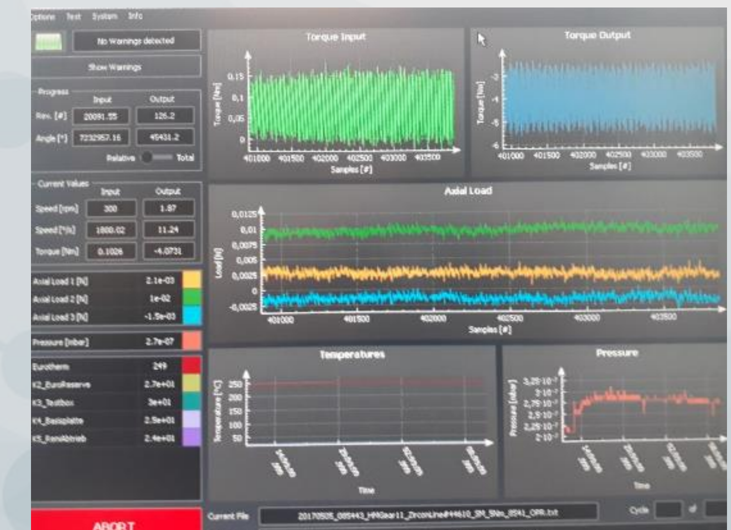


Thermo-mechanical behavior of new Gear Test Facility “HADES” between -150°Cand 300°C



Examples – Harmonic Drive Gears for Space

New Gear Test Facility "HADES" between in operation



Summary

Harsh space environment requires special testing for the qualification of materials used for satellites, spacecrafts, launchers etc.

AAC offers the infrastructure and experience to offer a wide range of qualification tests.

Thank you!

The ICARUS project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement No 713514.

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