Abstract for Presentation at the 2009 TMS Annual Meeting & Exhibition

Abstract Title: Advances in non-contact measurement of creep properties

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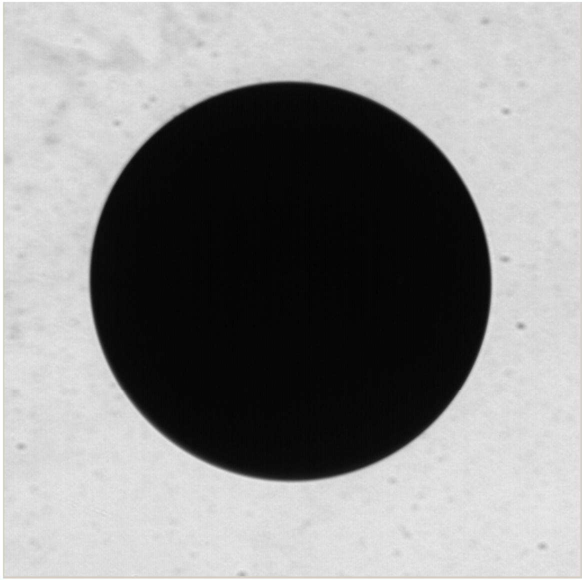
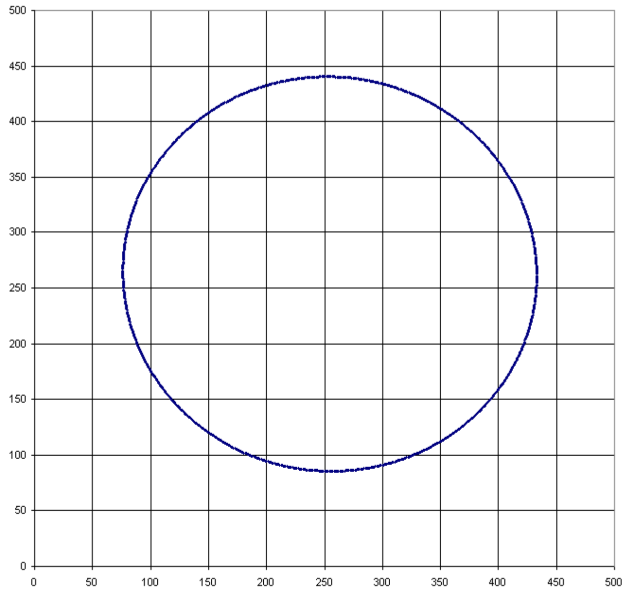
Speaker: Robert Hyers

Abstract: As the required service temperatures for superalloys increases, so do the demands on testing for development of these alloys. Non-contact measurement of creep of refractory metals using electrostatic levitation has been demonstrated at temperatures up to 2300 C using samples of only 20-40 mg. These measurements load the spherical specimen by inertial forces due to rapid rotation. However, the first measurements relied on photon pressure to accelerate the samples to the high rotational rates of thousands of rotations per second, limiting the applicability to low stresses and high temperatures. Recent advances in this area extend this measurement to higher stresses and lower-temperatures through the use of an induction motor to drive the sample to such high rotational speeds. Preliminary results on new measurements on new materials will be presented.

Advances in non-contact measurement of creep properties





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NASA MSFC ESL

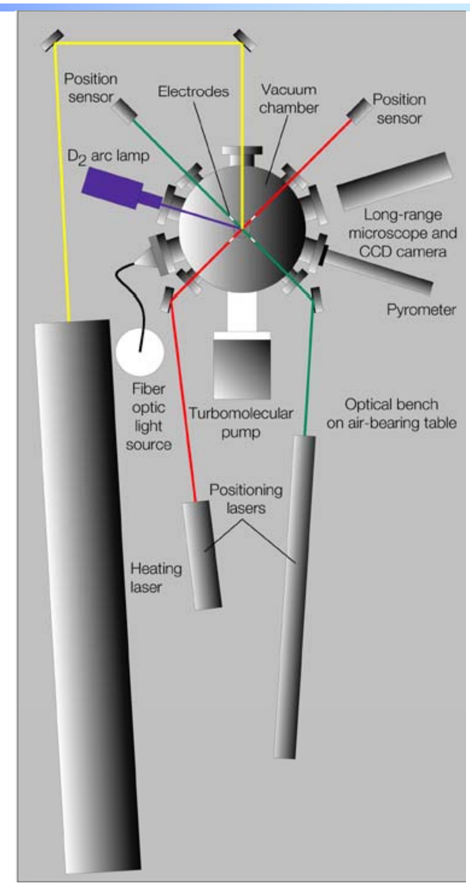
TMS Annual Meeting, February 16-19, 2009

Non-Contact Processing

- Fields support sample's weight:
  - Electromagnetic, Electrostatic, Acoustic, Aerodynamic, combinations.
- No container in contact with sample:
  - Reduced Contamination.
  - High temperature, >2500 ºC
  - Highly reactive samples.
  - Access to metastable phases, undercooling.
- Only optical access to samples.

ESL

- Processes or melts levitated samples
- Charged samples supported by electric field
- Provides a quiescent environment for the samples
- Metals, alloys, glasses, ceramics and semiconductors

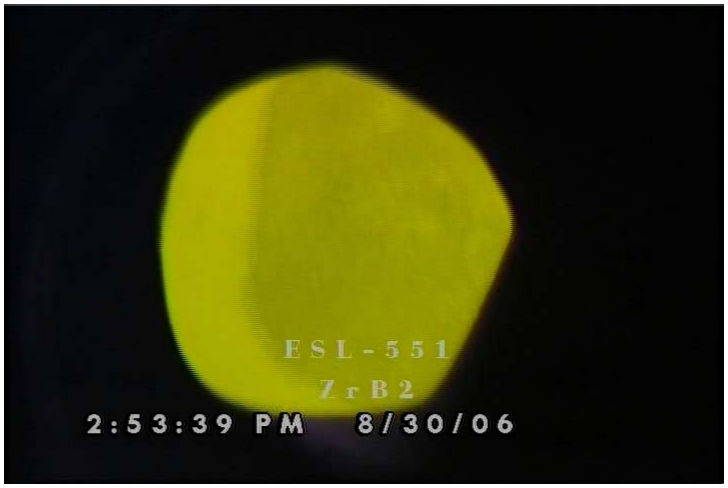


J.R. Rogers, MSFC

Materials Processed via ESL

Glass Melt with Crystallites

Metals and Alloys

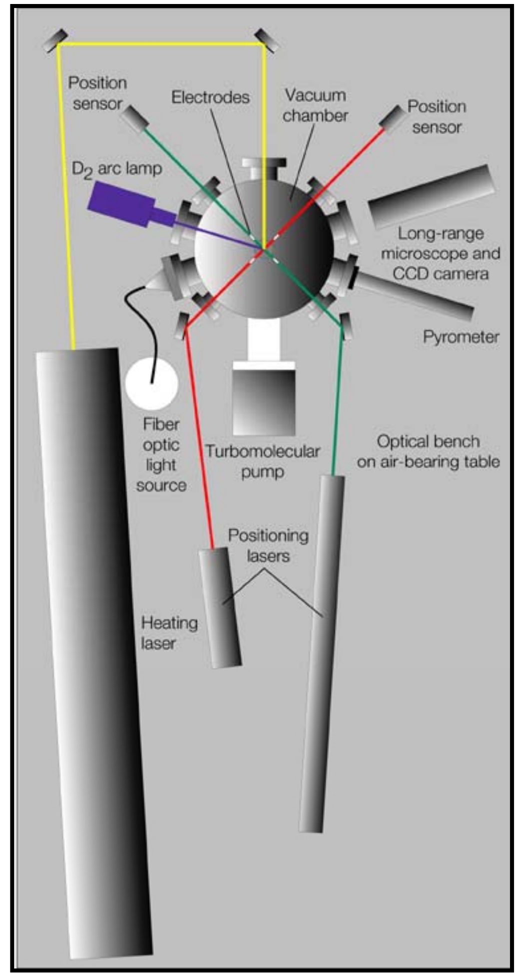


Ceramics, including pressed materials like ZrB_2

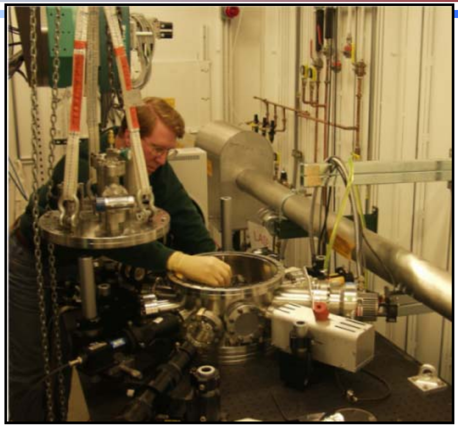
Other materials include polymers, semiconductors, solids, melts, and liquids.

MSFC ESL Facility

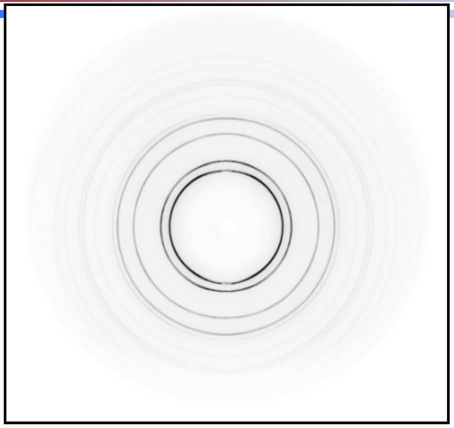
- The MSFC ESL Facility is a materials characterization facility that provides materials characterization data to users.
- Data files for thousands of melt cycles and hundreds of samples have been delivered to investigators, resulting in the development of new alloys, glasses, and numerous technical papers and journal articles.
- The MSFC ESL facility can provide measurements of thermophysical properties, which include creep strength, density and thermal expansion, emissivity, specific heat, and phase diagrams. For melts, viscosity and surface tension can be measured.
- Data can be obtained at ultra-high temperatures for materials being developed for propulsion applications.
- Samples: 2-3 mm diameter spheres (30-70 mg)
- Heated by lasers: 200W Nd:YAG or 300W CO_2



Phase Diagram Studies



Portable ESL at Argonne National Laboratory (ANL)

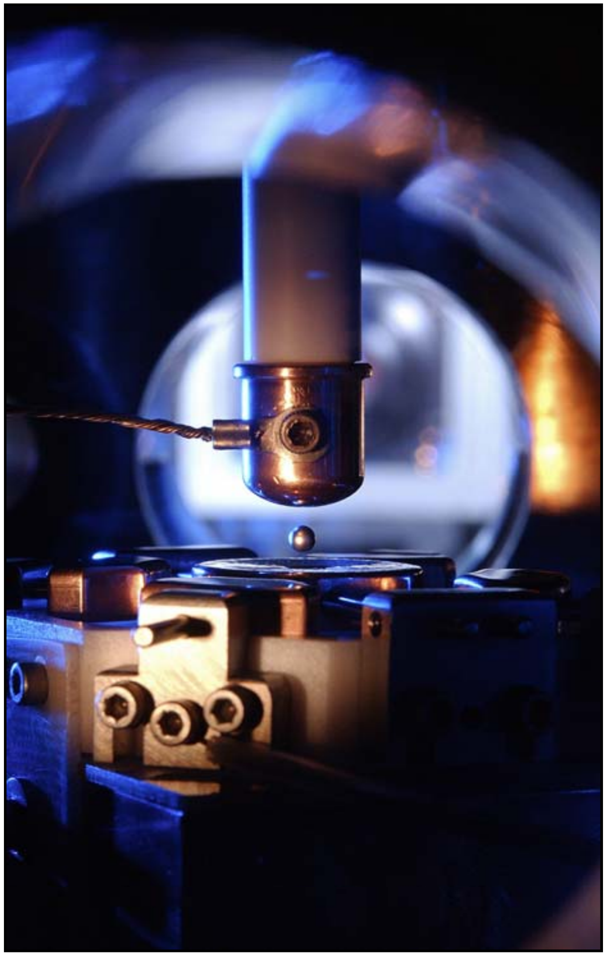


X-ray diffraction image from ESL processing at ANL

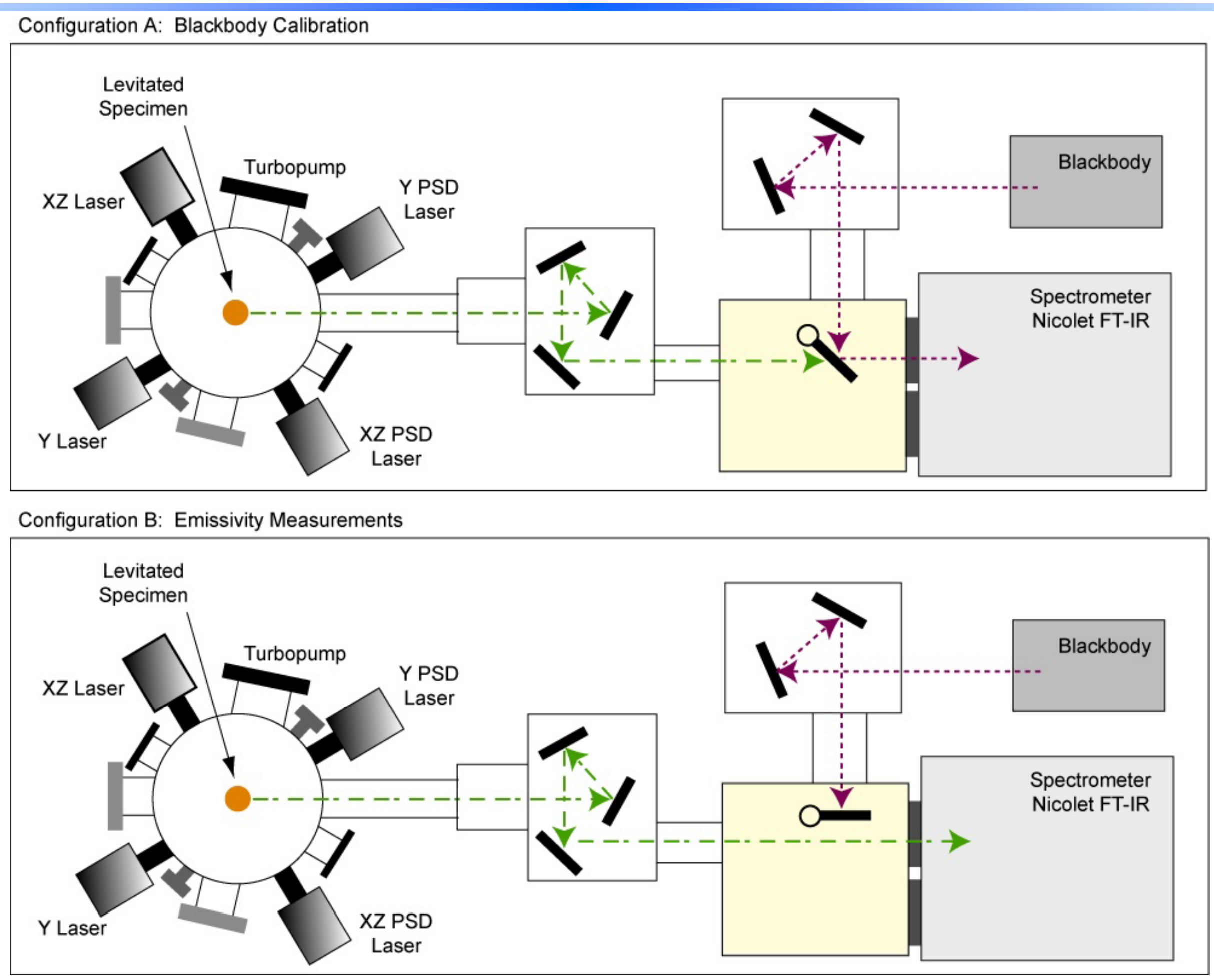
- Portable ESL used at the high-energy (125 -keV) synchrotron x-ray source at ANL.
- Provides *in-situ* determination of the atomic structures of equilibrium solid and liquid phases, including undercooled liquids, as well as real-time studies of solid-solid and liquid-solid phase transformations.
- Use of image plate (MAR345) or GE-Angio detectors enables fast (30 ms – 1s) acquisition of complete diffraction patterns.
- More rapid and accurate technique than conventional methods, which involve annealing and quenching (trying to preserve high-temperature structure) with subsequent room-temperature x–ray diffraction and electron microscopy studies

ESL Emissometer System

- Data needed for thermal design
- Emissometer developed by AZ Technology
- Temperature range: 700 to 3500 K
- FT-IR capabilities:
  - 0.400 to 28 μm
  - Emittance mode
  - Multiple scan ranges
  - Filtering for heating laser wavelengths
- Blackbody source operated at same temperature as sample with matched collection geometry
- Emittance data from sample and blackbody source integrated over spectral range
- Ratio provides measure of total hemispherical emittance.
- Preliminary tests with Inconel and stainless steel show good agreement with literature.



Schematic of System for Emissivity Measurements

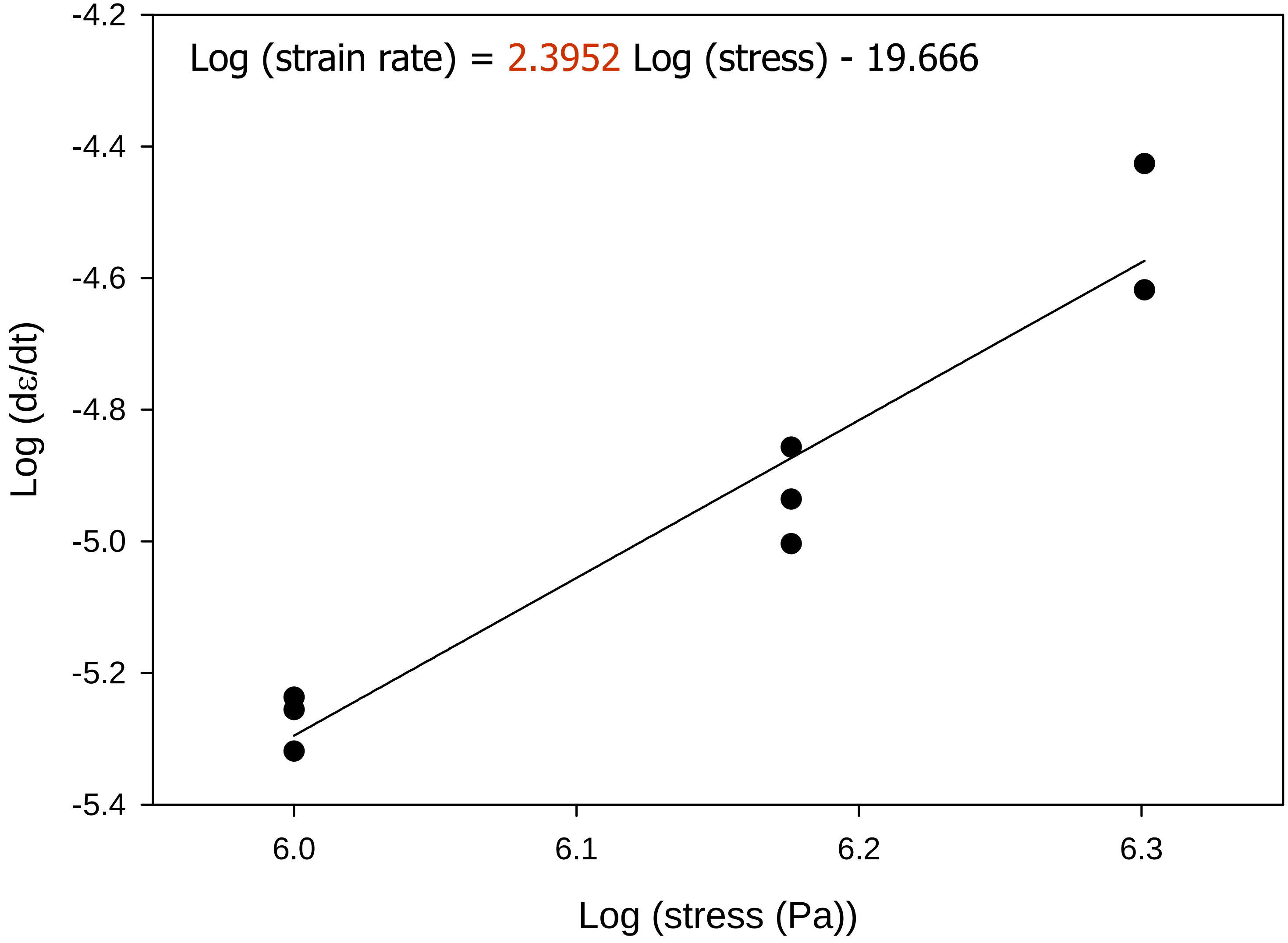


Emissivity Measurements with Levitated or Fixed Samples

Why Study Creep?

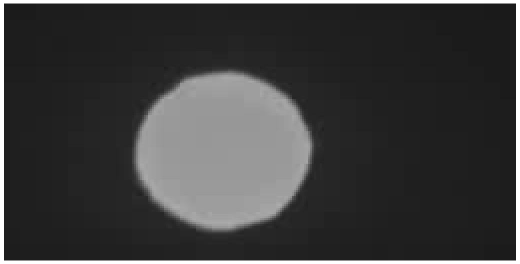
- Increasing needs for High-Temperature (HT) materials;
  - Hypersonic aircraft, non-eroding throats for solid rockets, next-generation jet engines;
- HT materials (T_m > 2000 °C) being developed;
  - Ultra-high temperature ceramics, Refractory Metals, and Niobium Silicides;
- Creep considered as one of the most important factors at high temperatures ($T > 0.5\ T_m$) ;
- Conventional methods for creep measurement limited to ~1700 °C;
- A new creep measurement method for HT materials strongly demanded.

Conventional Creep Testing

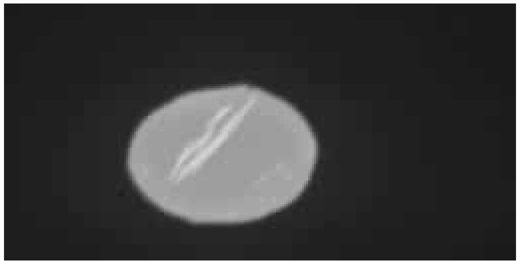


Non-Contact Creep Method

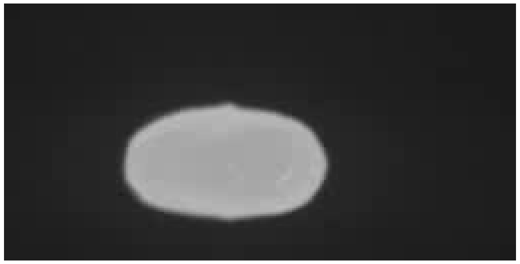
- Niobium Φ2.353 mm tested;
- Sample mechanically marked to facilitate rotation count;
- Sample levitated and stabilized in the vacuum chamber (~10^{-8} torr);
- Laser beam applied to heat up and rotate the specimen;
- Temperature maintained at 2,300 °C (T_m = 2,468 °C);
- Angular speed increased at ~870 Hz/hr and up to 3,200 Hz;
- Deformation noticed after 220 min;
- Sample dropped at 281 min;
- Machine vision software from density method used for data reduction and analysis.
- 6^{th} order Legendre Polynomials fitted to the extracted edges to define deformed shape;



220 min, Φ2.489 mm



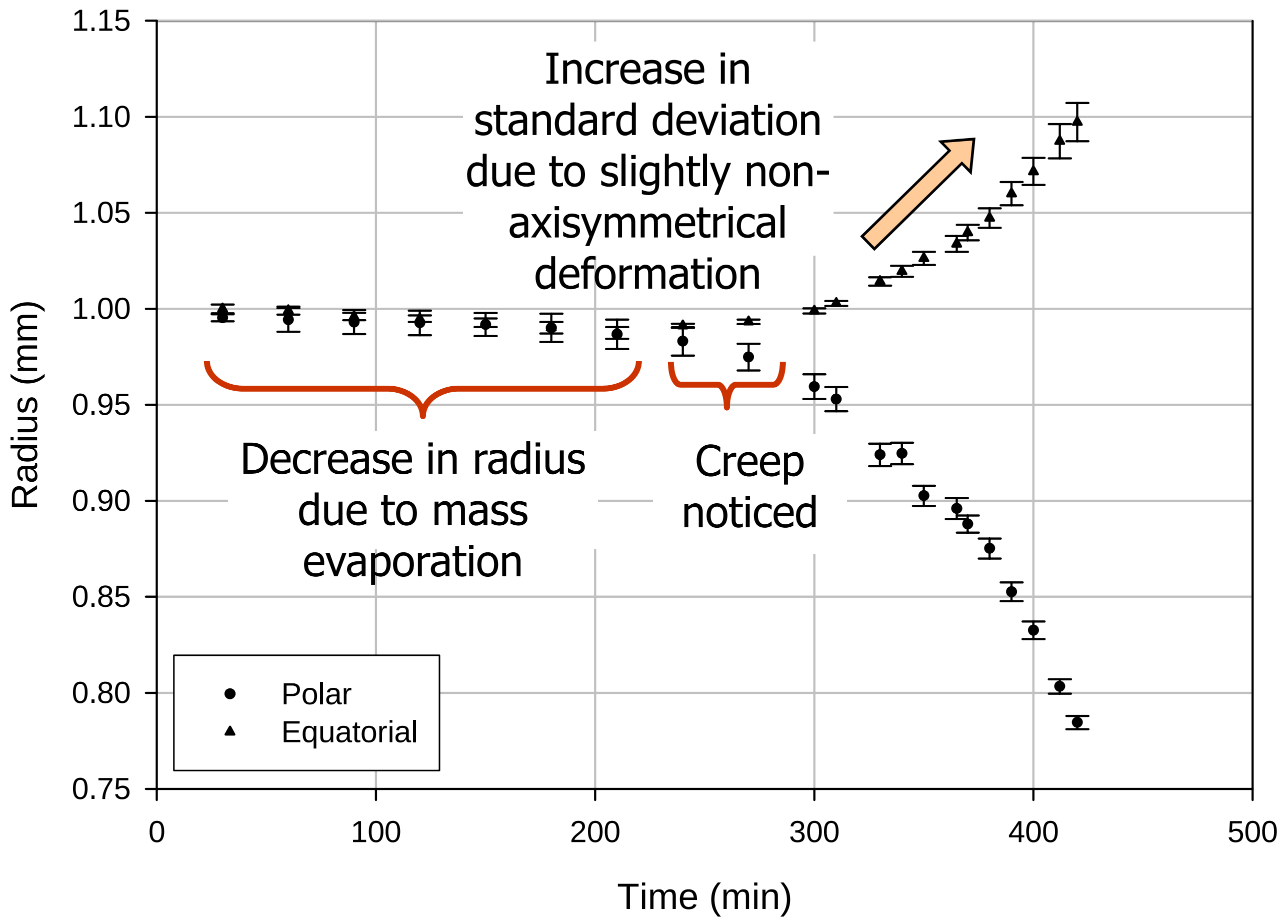
250 min, Φ2.692 mm

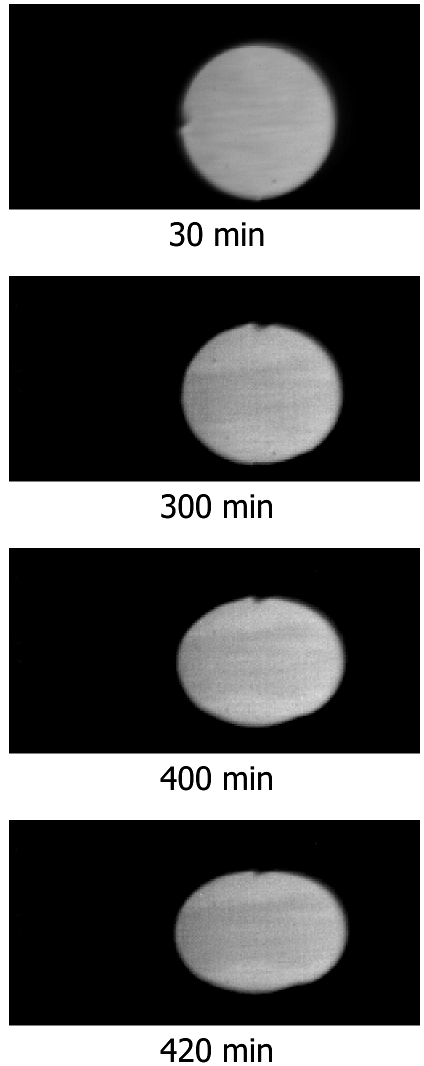


281 min, Φ3.005 mm

Rapidly Rotating Solid Spheres

Strain Ratio at $\varepsilon = 0.09$: 1.767





Creep Mechanism

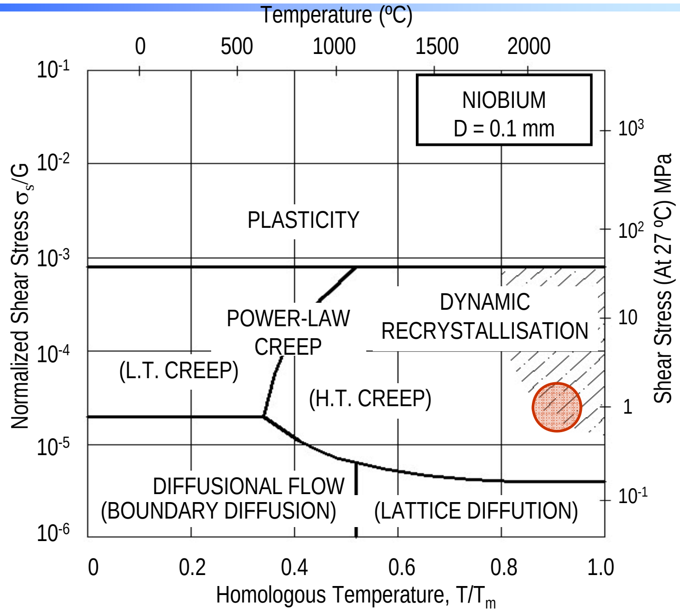
- Determination of constitutive relations for FE model;
- Maximum shear stress developed at the center of a rotating sphere:

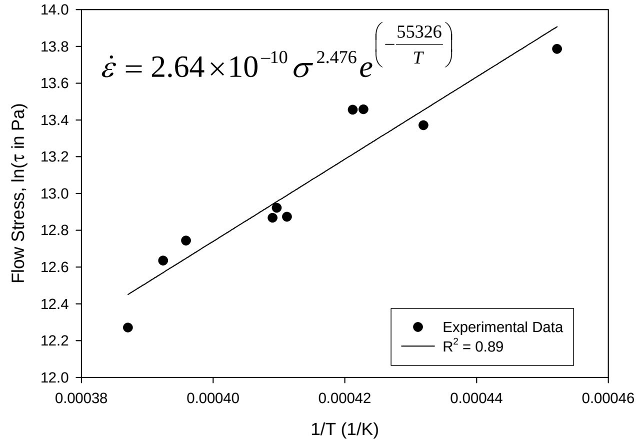
$$\tau(\omega) = 0.211\omega^2 r^2 \rho$$

  - Angular velocity: 20,106 rad/s;
  - Radius: 1.1765 mm;
  - Density: 8,562 kg/m^3;
  - Max shear stress = 1.01 MPa;

$$\dot{\varepsilon} = C\sigma^n e^{\left(-\frac{Q}{RT}\right)}$$

- Constants obtained by exp. Compared to results from the literature (Keissig *et al.*).



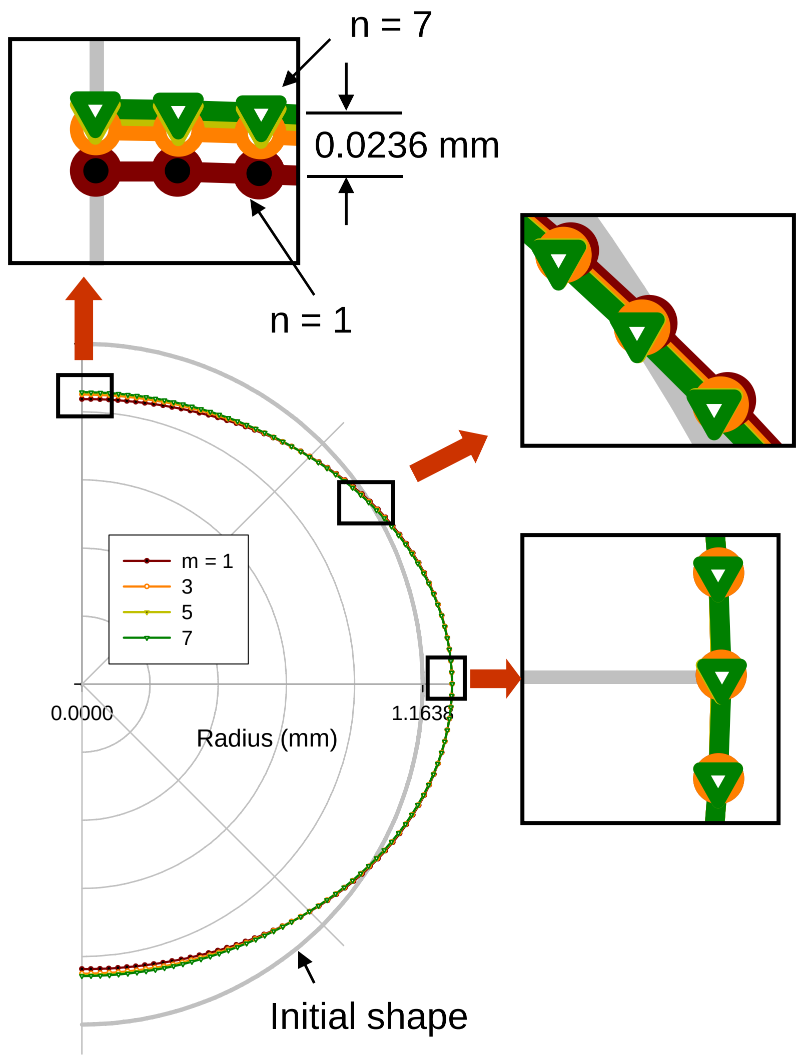


Stress Exponent and Deformed Shape

- FE models with different stress exponents run up to equatorial displacement of 0.1 mm (e_{eq} = 0.086);
- For the same equatorial strain, polar stain varies as a function of stress exponent;

$$\dot{\varepsilon} = C\sigma^n e^{\left(-\frac{Q}{RT}\right)}$$

- The higher the stress exponent, the less polar strain;
- Question:
  - Does the ratio of equatorial radius to polar radius give a **unique** stress exponent?
  - n ?= $f(R_{eq}/R_{pole})$



Stress Exponent and Shape

- 6th order Legendre Polynomials:

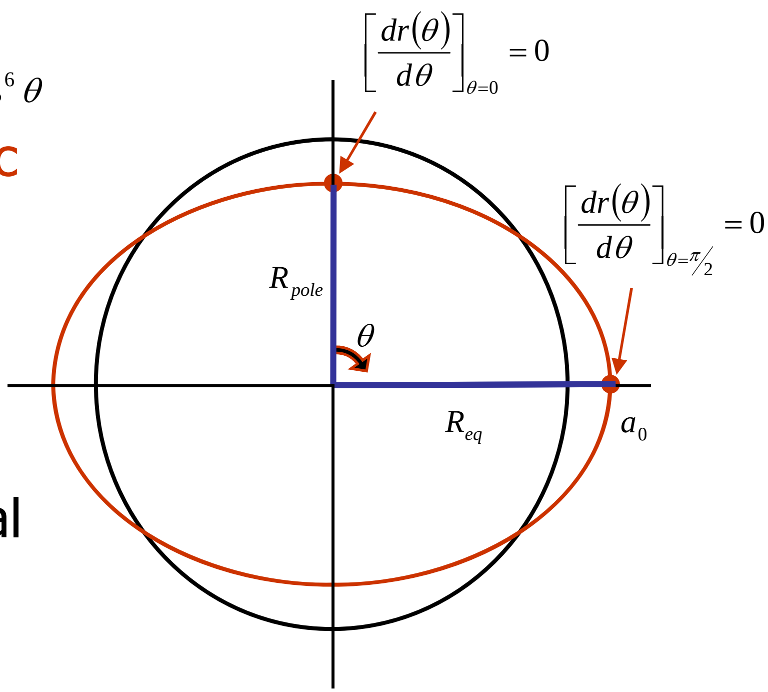
$$r(\theta)=\sum_{i=0}^{6} a_i P_i(\cos\theta)$$

$$r(\theta)=a_0+a_1\cos\theta+a_2\cos^2\theta+a_3\cos^3\theta+a_4\cos^4\theta+a_5\cos^5\theta+a_6\cos^6\theta$$

- Specimen assumed homogeneous, isotropic
  - Deforms axi-symmetrically
  - Also symmetric about the equatorial plane;
  - Odd terms can be neglected;

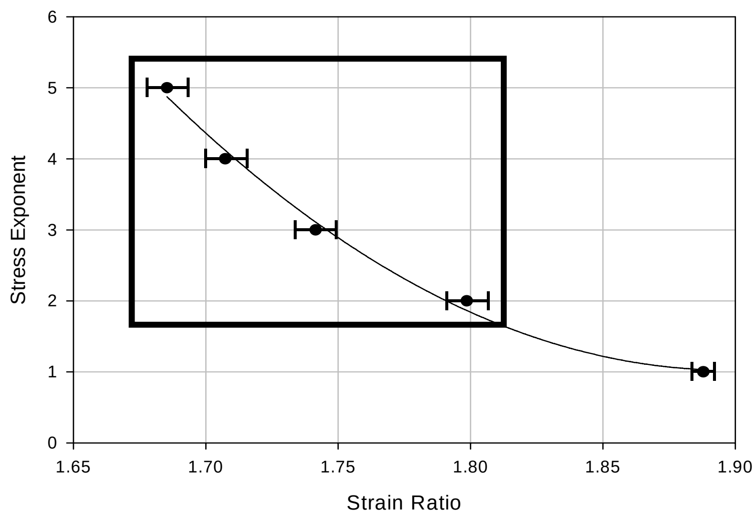
$$r(\theta)=a_0+a_2\cos^2\theta+a_4\cos^4\theta+a_6\cos^6\theta$$

- Four constants needed to define polynomial
  - 1st derivative at $\theta = \pi/2$ is 0;
  - Constant volume;
  - $P(\pi/2) = a_0$;
  - R_{eq}/R_{pole} (Radius Ratio),

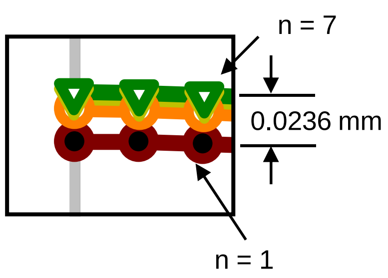


Sensitivity Analysis

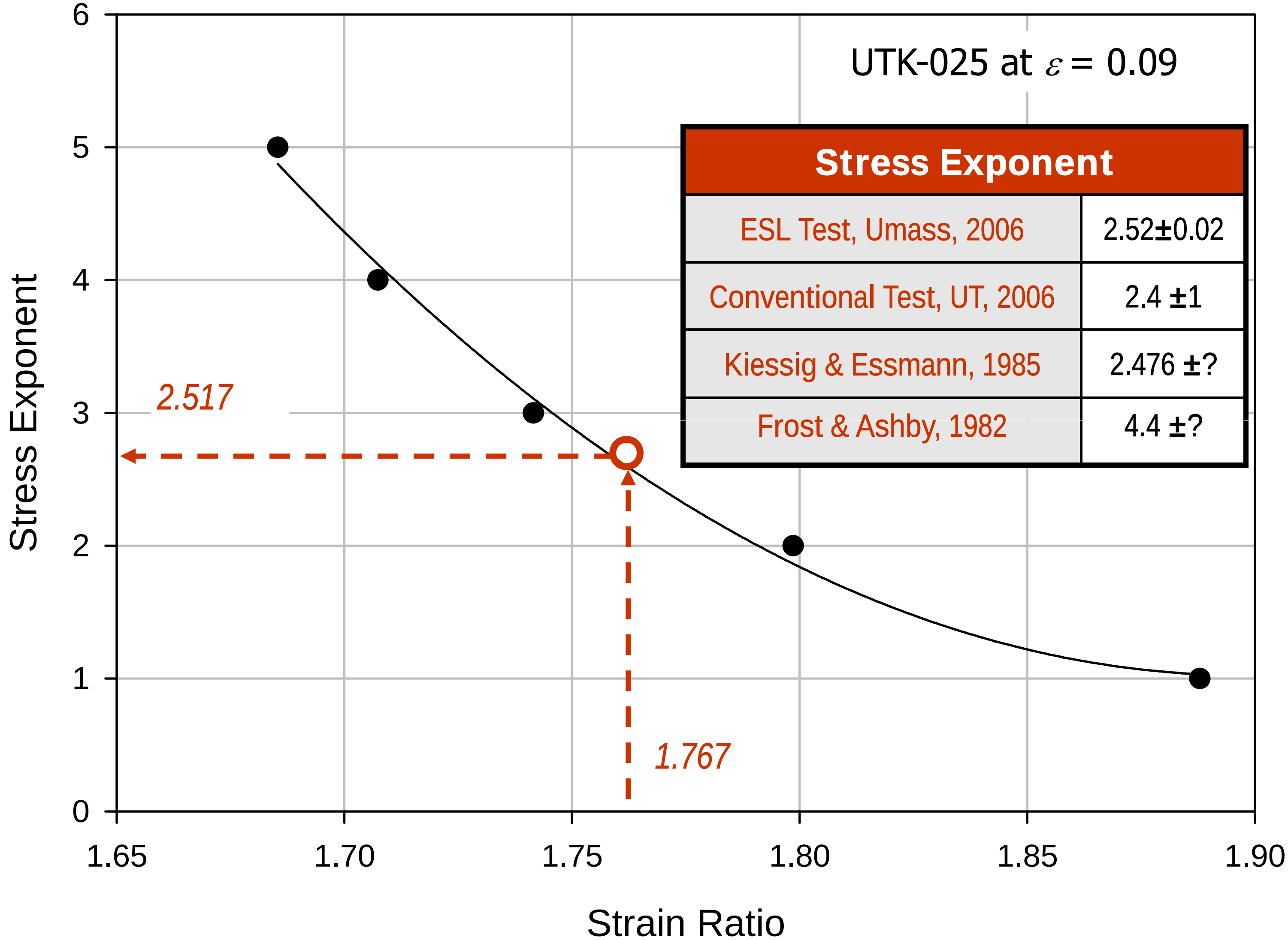
- Current measurement precision: 170 ppm in radius
- Stress exponent determined within ~1% for the most metals (2-5) at 0.09 strain;
- Accuracy increased by:
  - Using high-precision spheres;
  - Measurement at larger strains.



PPM	Error(%) Range	Stress Exponent				
		1	2	3	4	5
900	From	-0.18 %	-0.99 %	-0.89 %	-0.81 %	-0.75 %
	To	0.20 %	0.98 %	0.90 %	0.81 %	0.75 %
700	From	-0.14 %	-0.76 %	-0.70 %	-0.63 %	-0.58 %
	To	0.15 %	0.77 %	0.70 %	0.63 %	0.59 %
500	From	-0.10 %	-0.54 %	-0.50 %	-0.45 %	-0.42 %
	To	0.11 %	0.55 %	0.50 %	0.45 %	0.42 %
300	From	-0.06 %	-0.33 %	-0.30 %	-0.27 %	-0.25 %
	To	0.06 %	0.33 %	0.30 %	0.27 %	0.25 %
100	From	-0.02 %	-0.11 %	-0.10 %	-0.09 %	-0.08 %
	To	0.02 %	0.11	0.10 %	0.09 %	0.08 %



Result: Stress Exponent



Stress Exponent	
ESL Test, Umass, 2006	2.52±0.02
Conventional Test, UT, 2006	2.4 ±1
Kiessig & Essmann, 1985	2.476 ±?
Frost & Ashby, 1982	4.4 ±?

Non-contact Creep: Advantages and Limitations

- Non-contact => No temperature limit
- Stress exponent from a single test
- Metals, Ceramics, Semiconductors
- 2-3 mm diameter samples
  - Very little material required, but
  - Microstructural length scale matters!

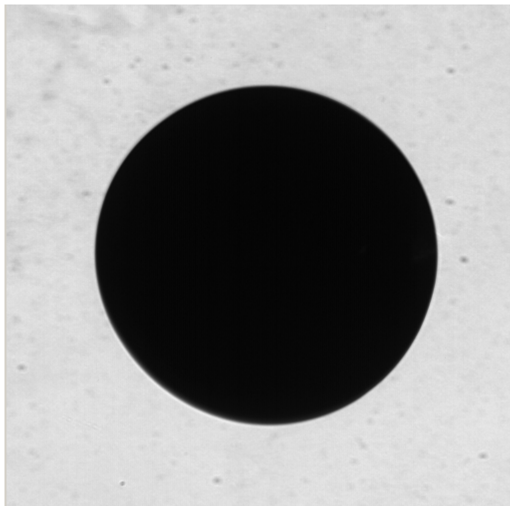
Ongoing Work for Creep

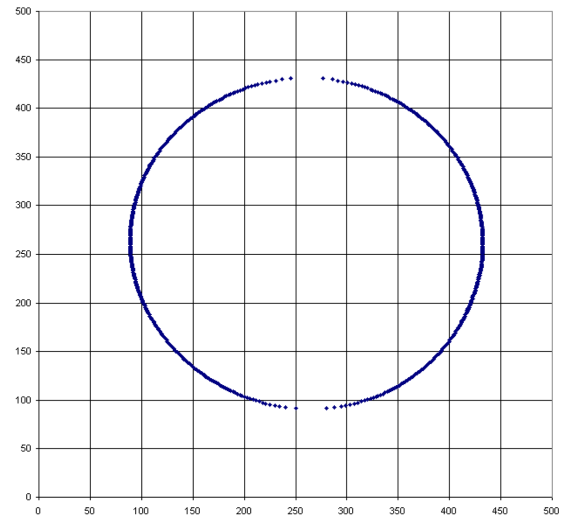
- New materials:
  - Nb superalloys, UHTC, refractory metals.
- Higher stresses:
  - Induction motor for sample rotation.
  - Enabling for measurements below ~2000°C.
- Expanding collaborations.

Conclusions

- ESL is a powerful tool for development of high temperature materials.
- No theoretical limit to temperature; demonstrated at 3400+°C.
- Measurements demonstrated include:
  - Phase determination
  - Emissivity
  - Creep
  - Density and Thermal Expansion

Acknowledgements





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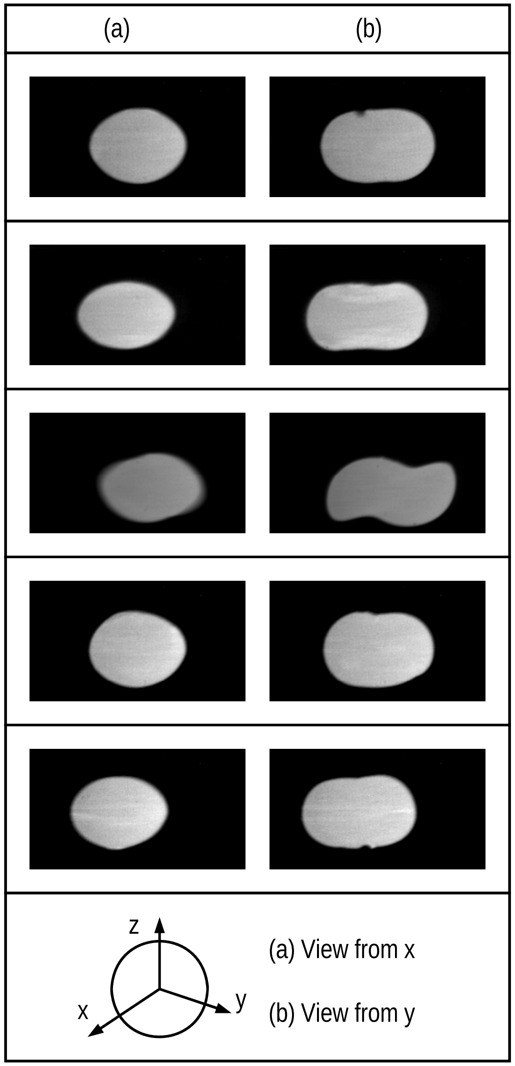
The End

Back-up Slides Follow

Experimental Challenges

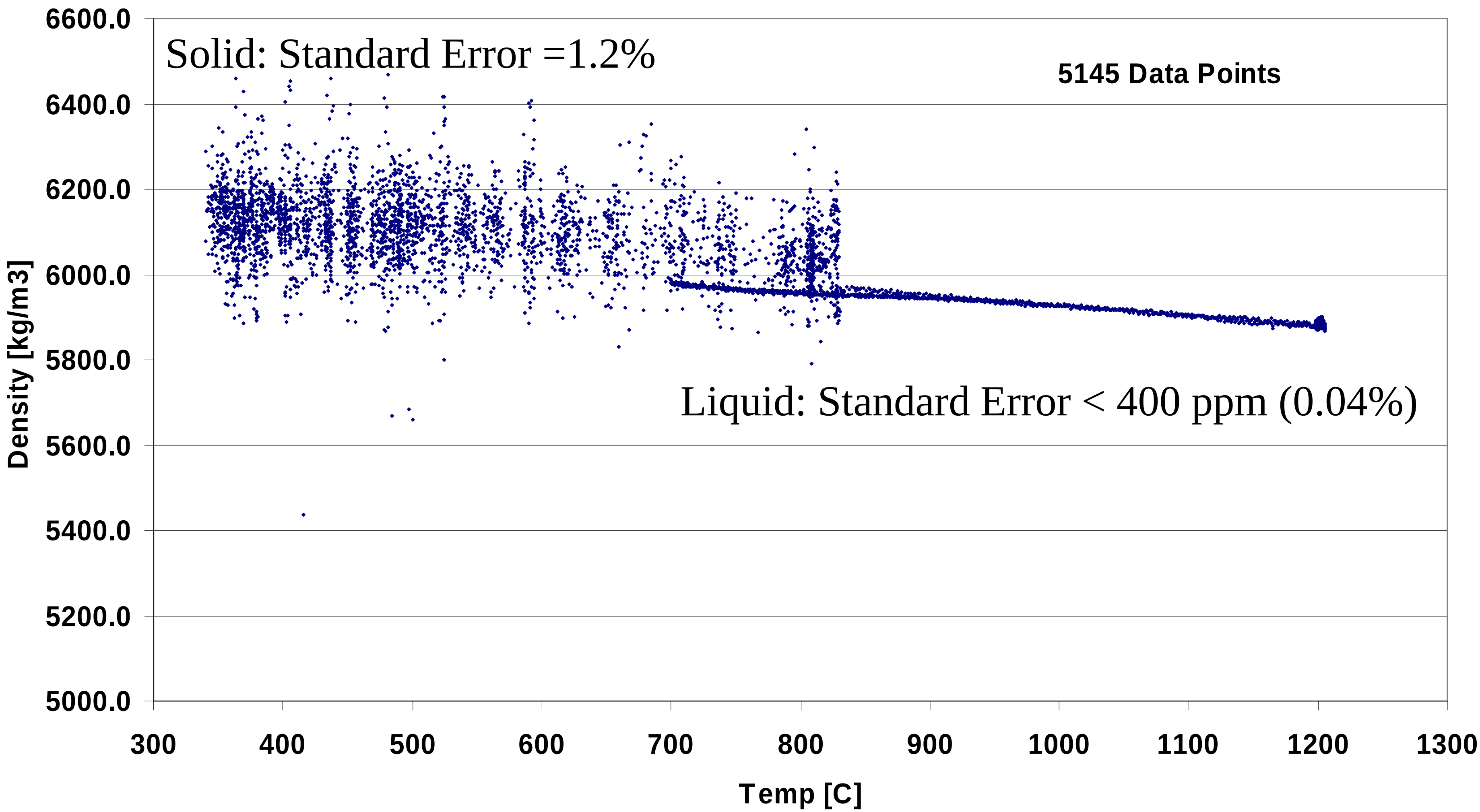
- Non-axisymmetrical deformation;
  - Due to poor quality of machined samples;
  - Hinders effective comparison to the numerical and analytical results;
- High precision Nb sphere used for validation;
  - Manufactured by Industrial Tectonics Inc., Dexter, MI;
  - 2.0 mm in diameter;
- Testing conditions;
  - Temperature set as 1,985 °C for the effective comparison to the conventional creep test;
  - The same testing procedure applied.



Other Measurements

- Specific Heat, Enthalpies of Transformation (Rhim, Kelton, Johnson, others).
- Optical properties: spectral and total hemispherical emissivity.
- Electrical Conductivity?
- Solidification Rate / Transformation Rate
- Phase Selection
- Nucleation Rate

$Ti_{39.5}Zr_{39.5}Ni_{21}$ Density



Non-Contact Measurement of Density

- Ti-Zr-Ni alloys are very reactive:
  - Containerless method
  - High vacuum ~10^{-7} Torr
  - High maximum temperature (~1600 ºC)
- Electrostatic Levitation (ESL)
  - NASA MSFC ESL Facility
- Video method for density
  - High data rates: 25 frames/sec @ 512 x 512 x 8-bit
    6.5 MB/sec = 190 GB/8 hours = 1 TB/work week
  - Requires automated data reduction and analysis.
  - Precision and accuracy?

Verification and Testing

- Simulated images
  - Recover the input parameters?
  - Record precision and accuracy of automated analysis
- Test Cases
  - Sigmoid intensity profile
    - Test sub-pixel interpolation
  - Legendre silhouettes
    - Test vector fit, parameter optimization
  - Translating image: move Legendre silhouette 100 steps/pixel
    - Test sensitivity and quantization

Calibration

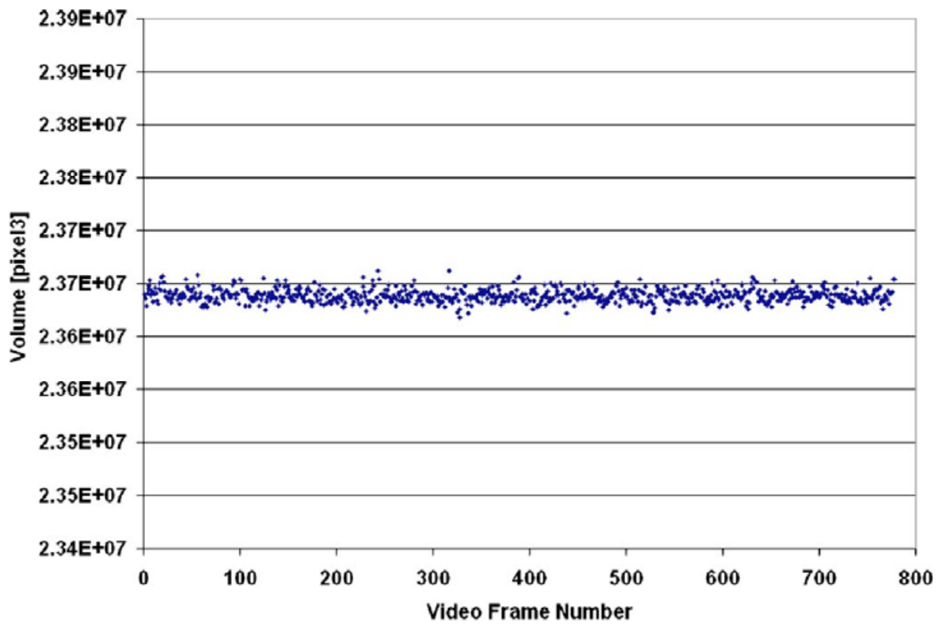


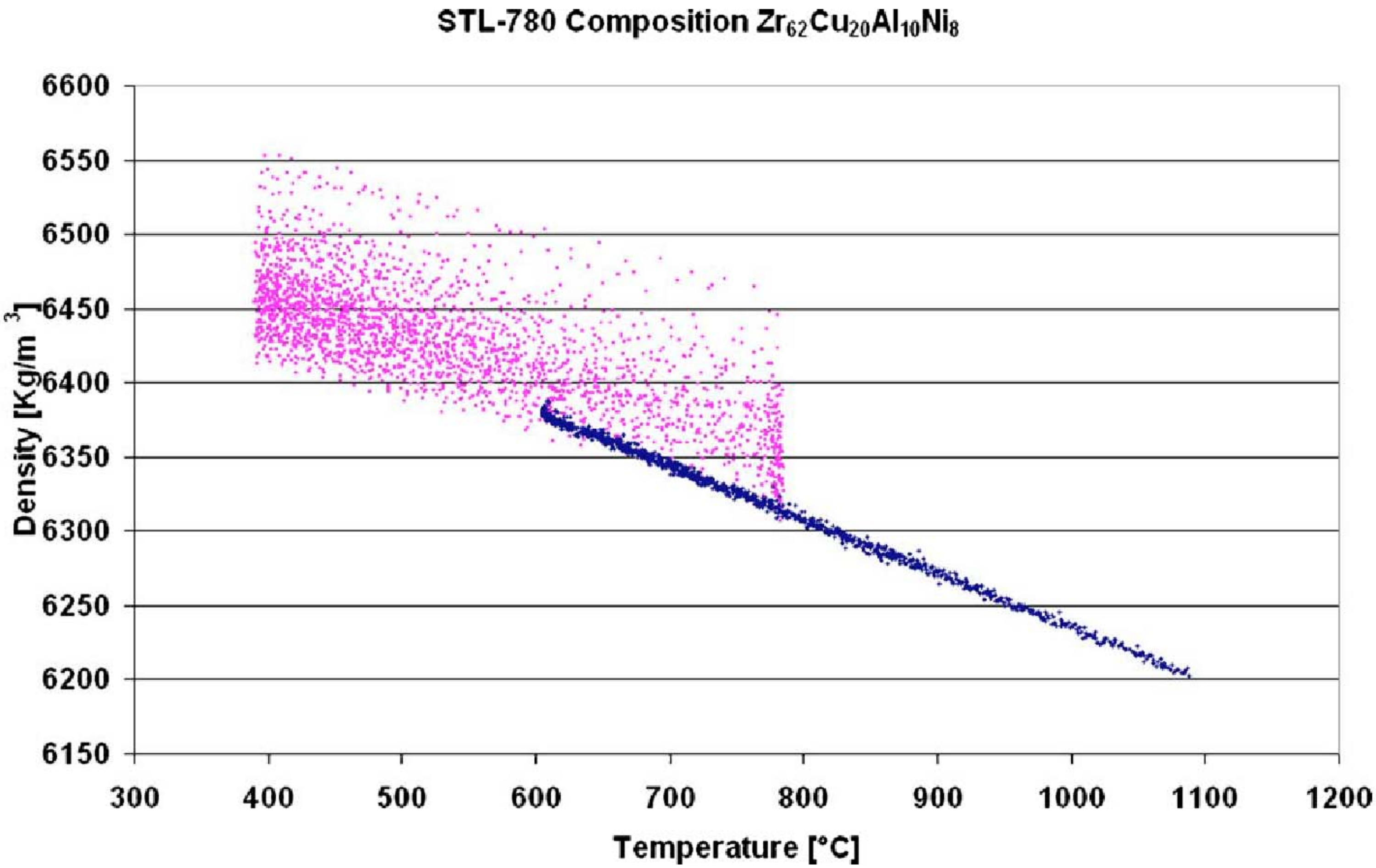
FIG. 9. (Color online) Calibration sphere analysis plot of volume vs frame number. The standard error, normalized by the average volume, is 0.0265%.

- Calibration with Grade 3 WC-Co spheres (Tolerance: 75 nm sphericity, 750 nm diam).
- Precision about 250 ppm (0.025%).
  - Standard deviation of multiple observations of same sample
- Accuracy about 100 ppm (0.01%).
  - Comparison of density of samples 2.0 – 2.5 mm diameter.
  - Average of about 200 observations on each sample.

Density Calculation

- Least Squares fit a 6th order Legendre polynomial to edge points.
- Integrate polynomial to get volume via calibration factor.
- Calibration gives volume in m^3 instead of $pixels^3$.
- Mass measured on microgram balance.
- Thousands of data points allow detailed statistical analysis of uncertainty in density, thermal expansion vs. temperature.

Results



Standard Errors: Liquid = 0.038% (380 ppm), Solid = 0.430% (4300 ppm)

More Results

5 weeks of experiments on 19 Samples

1.5 TB of Videos

2 years of process development

Yields:

Results:

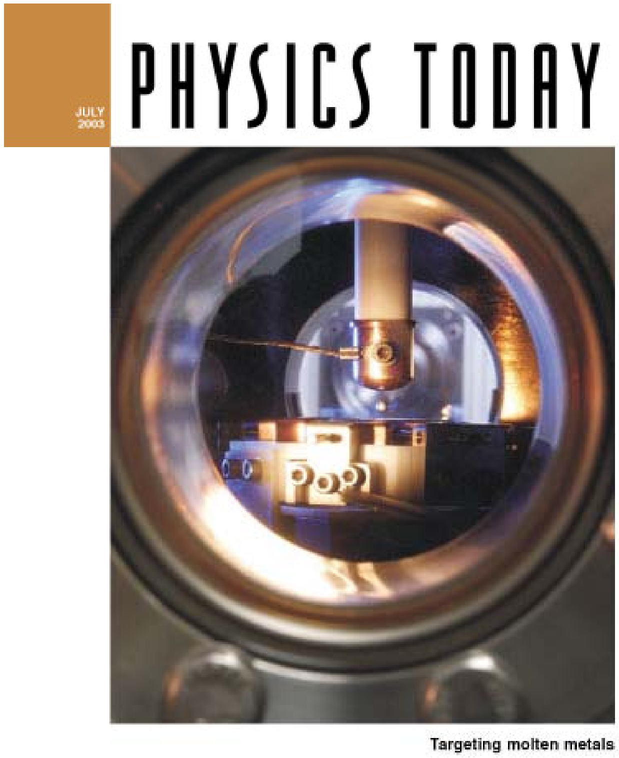
Linear fits with 95% confidence intervals

Table 1: Linear fits for density of assorted Ti, Zr and Fe alloys in the liquid state. The form of the fit is $\rho(T) = A + B(T - T_{ref})$ [Kg/m^3].

Composition	A	+/-	B	+/-	Tref °C	Std. Err %	R^2	T Range °C
$Ti_{39.5}Zr_{39.5}Ni_{21}$	5956.84	1.91	-0.168	0.002	810	0.05	0.97	700-1130
$Ti_{35}Zr_{35}Ni_{30}$	6219.83	19.47	-0.303	0.022	850	0.41	0.54	810-1070
$Fe_{50}Cu_{50}$	7089.03	17.55	-0.693	0.018	1088	0.22	0.95	830-1170
$Ti_{39.5}Zr_{39.5}Ni_{21}(Ag_2)$	6087.33	5.92	-0.309	0.007	820	0.07	0.97	785-1040
$Ti_{39.5}Zr_{39.5}Ni_{21}(Pt_2)$	6173.00	8.53	-0.325	0.009	860	0.18	0.92	795-1180
$Ti_{30}Zr_{30}Ni_{40}$	6335.68	9.24	-0.359	0.011	910	0.28	0.86	720-1120
$Ti_{37}Zr_{42}Ni_{21}$	6013.28	10.70	-0.337	0.012	810	0.07	0.71	710-1200
$Ti_{39.5}Hf_{39.5}Ni_{21}$	9060.10	8.43	-0.421	0.008	955	0.13	0.96	830-1350
$Ti_{39.5}Zr_{19.75}Hf_{19.75}Ni_{21}$	7470.54	12.05	-0.382	0.012	850	0.2	0.91	810-1250
$Ti_{39.5}Zr_{35.55}Hf_{3.95}Ni_{21}$	6237.33	5.80	-0.316	0.007	820	0.2	0.84	735-1150
$Ti_{50}Zr_{50}$	5458.45	4.43	-0.203	0.005	1545	0.32	0.62	1245-1670
$Ti_{73}Fe_{21}Si_6$	4862.88	6.14	-0.310	0.006	1025	0.07	0.95	965-1190
$Zr_{57}Ti_5Ni_8Cu_{20}Al_{10}$	6363.50	1.21	-0.376	0.001	775	0.05	0.99	640-1010
$Zr_{67}Pd_{33}$	7597.07	5.62	-0.395	0.005	1085	0.14	0.96	755-1265
$Ti_{35.5}Zr_{35.5}Ni_{21}Si_8$	5844.56	14.86	-0.343	0.012	910	0.22	0.92	1030-1485
$Ti_{37.5}Zr_{37.5}Ni_{21}P_4$	5947.82	5.62	-0.310	0.005	816	0.13	0.97	900-1450
$Ti_{37.5}Zr_{37.5}Ni_{21}Si_4$	5927.83	2.48	-0.299	0.002	816	0.1	0.99	780-1395
$Ti_{38.5}Zr_{38.5}Ni_{21}P_2$	5952.83	2.73	-0.301	0.003	810	0.08	0.99	750-1220
$Ti_{38.5}Zr_{38.5}Ni_{21}Si_2$	5911.22	2.90	-0.312	0.003	813	0.07	0.99	740-1135

Structure of Liquid

- Combine ESL with high-energy x-ray diffraction: Beamline ESL (BESL)
- High-energy photons:
  - penetrate sample: bulk structure, not surface.
  - small angle:large range of q.
- High intensity synchrotron source: rapid acquisition, transient analysis.
- Simultaneous collection of data on thermophysical or thermomechanical properties.



With Ken Kelton (PI), Jan Rogers, Alan Goldman, Doug Robinson, et al.