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TITLE CREEP SPECIMENS RECEIVED **PROJECT** CREEP DEFORMATION STUDIES

Continued From Page

Date Received	Specimen Id.	Grade	Stress (Ksi)	Temp. (F)
11/2/2007	G00565	5	133.43	302
	G00564	5	139.495	302
	G00566	5	127.365	302
	G00567	5	121.300	302
	G00568	5	103.105	302
	G00569	5	84.910	302
	G00570	5	66.715	302
	G00571	5	48.520	302
11/8/2007	G00572	5	124.695	RT
	G00573	5	118.958	122
	G00574	5	109.735	212
	G00575	5	91.460	482
	G01027	5	118.958	122
	G01028	5	97.920	392
	G01029	5	97.920	392
11/9/2007	G00553	7	34.980	302
	G00554	7	33.390	302
	G00556	7	27.030	302
	G00552	7	36.570	302
	G00558	7	17.490	302
	G00557	7	22.260	302
	G00555	7	31.800	302
	G00559	7	12.720	302
11/14/2007	G00560	7	44.668	RT
	G00561	7	41.395	122
	G00562	7	34.425	212
	G00563	7	21.250	482
	G01024	7	21.250	482
	G01025	7	23.418	392
	G01026	7	23.418	392

Continued To Page

SIGNATURE [signature] DATE 11/19/2007

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~~PROPRIETARY INFORMATION~~ 6/22/10 [initials]

TITLE TEM SAMPLE PREPARATION **PROJECT** CREEP DEFORMATION STUDIES

Continued From Page

1. Sample ID: 600560

a) Cutting the specimen:

The Grade 7 Titanium specimen deformed at 85% yield stress and at room temperature (25°C) is cut using Buehler Isomet low speed saw. The saw is partially submerged in Buehler Isocut Fluid to allow lubrication while cutting the metal. An initial cut is made to reveal the specimen interior. Next, thin slices of the specimen are cut by moving the sample with respect to the saw. A specimen thickness of 120 μm is cut by dialing the micrometer attached to the saw by 18 divisions. Seven such specimens are cut initially.

Following that, a Gatan disk punch is used to produce a 3mm diameter disk from the cut slices.

b) Dimpling of the specimen:

The dimpling of the specimen is carried out using South Bay Technology Dimpler. A specimen holder is initially heated over a hot plate (make: VWR Scientific). A small amount of mounting wax (Quickstick 135) is applied at the center of the specimen stage. The wax is allowed to melt before placing the specimen over it. After centering the specimen over the specimen holder, the setup is allowed to cool down so that the specimen is glued to the center of the specimen holder. Once cooled, the stage is placed onto the dimpler underneath the polishing wheel. A small amount of 6-μm diamond paste is put on top of the specimen and on the polishing wheel. The specimen is dimpled by switching on the apparatus and continued until the entire surface ~~is~~ appears dimpled (a dark circle which nearly covers the 3mm sample disk). The sample is then flipped over and the process is repeated on the other side.

Such a dimpling procedure was followed for all the seven specimens.

c) Electro-jet polishing of the specimen:

The final thinning of the sample was performed by electro-jet polishing. Jet polishing is a chemical process that uses a combination of an acid containing electrolyte and electric

Continued To Page 3

SIGNATURE [signature] DATE 11/26/2007

DISCLOSED TO AND UNDERSTOOD BY DATE

~~PROPRIETARY INFORMATION~~ 6/22/2010

TITLE TEM SAMPLE PREPARATION (CONT.) **PROJECT** CREEP DEFORMATION STUDIES

Continued From Page

current to remove material from the sample. The sample was thinned using a twin-jet polishing unit. The polishing solution used for the jet polishing consists of 3% hydrochloric acid (HCl), 3% sulfuric acid (H_2SO_4) and 94% methanol (CH_3OH). The dish containing the polishing solution is placed in a methanol bath and cooled with dry ice so that the samples are polished in the range of 60°c to 70°c at 20V.

Procedure for TEM sample preparation by Jet-Polishing

1. In the fumehood: in large, round Pyrex dish, place 120ml methanol.
2. Slowly add 4ml sulfuric acid to the Pyrex dish and stir to mix.
3. Slowly add 4ml hydrochloric acid to the solution and stir to mix.
4. Pour methanol into rectangular Styrofoam container to depth of approximately 0.75".
5. Place the Pyrex dish with the electropolish solution into the Styrofoam container with caution.
6. Add small pieces of dry ice to the methanol bath with caution (not to let the ice fall inside the electropolish solution).
7. Place the electropolishing equipment into the Pyrex dish such that the pumps are submerged into the solution.
8. Place the dimpled TEM specimen into the specimen holder of the electropolishing equipment.
9. Connect the anode to the specimen and make sure that the specimen is in the path of the jets. Turn on the power switch.
10. Turn the polish dial to maximum of 10 to begin the process.
11. Turn the speed dial to maximum as well.
12. Turn off the power after every minute to check if a tiny hole is created due to the jet polishing. Continue the process otherwise.
13. Stop the electropolishing when there is a "pin-hole". Look for the hole by holding the TEM specimen against a bright light source. Discard the specimen if the hole is too big.
14. Rinse and dry the specimen once the sample appears to be satisfactory for TEM analysis. Follow the cleaning with ultrasonic cleaning by acetone.
15. Clean the setup after use.

Note: Seven TEM specimens are made which have a "pin-hole".

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SIGNATURE [signature] DATE 11/27/2007

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~~PROPRIETARY INFORMATION~~ 6/22/10

TITLE CREEP SPECIMENS RECEIVED **PROJECT**

Continued From Page

Date Received	Specimen ID.	Grade	Stress (ksi)	Temperature (°F)
12/04/07	F37100	7	60.433	RT
	F37101	7	57.805	RT
	F37102	7	55.178	RT
	F37103	7	52.550	RT
	F37104	7	44.668	RT
	F37105	7	36.785	RT
	F37106	7	28.903	RT
	F37107	7	21.020	RT
	F37108	7	46.663	212
	F37109	7	44.605	212
	F37110	7	42.578	212
	F37111	7	40.550	212
	F37112	7	34.468	212
	F37113	7	28.385	212
	F37114	7	22.303	212
	F37115	7	16.220	212
	F37116	7	28.750	482
	F37117	7	27.500	482
	F37118	7	26.250	482
	F37119	7	25.000	482
	F37120	7	21.250	482
	F37121	7	17.500	482
	F37122	7	13.750	482
	F37123	7	10.000	482

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DATE 12/05/2007

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~~PROPRIETARY INFORMATION~~ 6/22/10

TITLE TEM SAMPLE PREPARATION **PROJECT**

Continued From Page

1. SAMPLE ID: F37112

TEM samples of Grade 7 Titanium alloy, deformed at 85% yield stress and at 100°C are prepared using the same technique ~~to~~ described before. In total, 7 TEM specimens are prepared using the procedure mentioned in Pages 2 and 3.

2. ~~TEM~~ SAMPLE ID: G00556

Seven TEM samples of Grade 7 Titanium alloy, deformed at 85% yield stress and at 150°C are prepared using the procedure described in Pages 2 and 3.

Continued To Page

SIGNATURE [signature] DATE 12/14/2007

DISCLOSED TO AND UNDERSTOOD BY DATE

TITLE Tensile Data Analysis of Gr 7 PROJECT

Continued From Page

Tensile curves of Grade 7 alloy at different temperatures are plotted using Microsoft Excel.

(1) The yield stress (0.2% Yield stress) decreases linearly with temperature.

This aspect is noticed during both Phase I & Phase II studies.

(2) There is a drop in the yield stress after the initiation of plastic deformation. This feature is more noticeable at temperatures greater than or equal to 100°C (212°F). The relative drop in the yield stress i.e. magnitude of the yield stress drop divided by the upper yield stress increases linearly with temperature.

The equation that 0.2% yield stress vs temperature curve follows is:

$$Y.S.\ (MPa) = -0.909\,T + 376.53;\ T = T(^\circ C).$$

The temperature is in degree centigrade.

Continued To Page

SIGNATURE [signature] DATE 12/17/2007

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TITLE TENSILE DATA ANALYSIS of GRADE 5 PROJECT

Continued From Page

Tensile curves (i.e. stress vs strain) of Grade 5 alloy are plotted at different temperatures (25°C, 50°C, 100°C, 150°C, 200°C and 250°C).

The curves are plotted to a strain of 1.0%. There is no hardening of the stress-strain curves until then (or a drop in the yield stress).

A plot of 0.2% yield stress versus temperature show the yield stress decrease linearly with temperature.

The equation that the curve follows is:

$$Y.S.\,(MPa) = -1.1485\,T + 1021.6\ ; \quad T = T(°C).$$

(Note: this equation is derived by combining the tensile data from both Phase I & II studies.)

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TITLE TEM ANALYSIS OF GRADE 7 **PROJECT**

Continued From Page

Sample ID: G00560 (GRADE 7)

A JOEL 2100 LaB_6 is used for TEM. The operating voltage is 200 kV.

The sample was creep deformed at 85% yield stress and at room temperature (25°C).

The sample showed a significant number of deformation twins and dislocations upon initial viewing.

The twins are characterized by wedge-shape (or parallel lines) within a grain.

Dislocations, on the other hand, appear as sharp lines (i.e. atomically sharp).

As the TEM sample is tilted by a small amount, it is observed that the bend contours appearing on the sample surface move alongwith. This is in contrast to the ~~distoc~~ dislocations or twins which do not immediately start moving across the sample (i.e. the dislocations remain fixed in its location where as the bend contours move). This aspect or observation is important when analysing sample defects. Also, bend contours are thicker than the dislocations.

Three (3) specimens of the same type were analysed for uniformity in the results. All the specimens showed twins and dislocations.

One of the specimen, however, had to be ion-milled as the edges near the hole appeared to be thicker and impeded our analysis.

As the observations of dislocations & twins were found on the other specimens as well (not ion-milled), it is concluded that additional ion milling may at times be employed for better TEM analysis.

Continued To Page

SIGNATURE DATE 12/21/2007

DISCLOSED TO AND UNDERSTOOD BY DATE

TITLE TEM of Grade 7 (G00560) PROJECT

Continued From Page 8

DISLOCATIONS

Slipping of dislocations were primarily observed on prism $\{1\bar{1}00\}$ planes and having Burgers vector $\vec{b} = \frac{1}{3}\langle 11\bar{2}0\rangle$

The deduction was made by two-beam condition in TEM and taking dark-field images of the dislocation region. The Burgers vector is determined through $g \cdot b$ analysis.

The line direction of the dislocation was also determined with the help of stereographic projection. By knowing the Burgers vector and the line direction, the slip plane was found and therby the nature of the dislocation (edge, screw, mixed).

In our case, the dislocation was found to be of screw type.

In order to determine if the screw dislocation $\frac{1}{3}\langle 11\bar{2}0\rangle$ was gliding on $(1\bar{1}00)$ or (0001) i.e. the prism or basal plane, bright field images were taken along these two zone axis. A dislocation gliding on $(1\bar{1}00)$ would appear as a single dislocation when observed along the $\langle 0001\rangle$ zone axis, wheras, the same dislocation feature would appear in stacks when observed along ~~<11$\bar{2}$0> zone axis~~. any other zone axis.

An analysis of this nature revealed that in the current case, the dislocations were gliding along the prism $(11\bar{2}0)$ - plane.

TWINS

Twins were initially recognized by their shapes in the TEM image. For further study, the nature of the twins, diffraction patterns were taken from the matrix and the twinned region. Furthermore (importantly), diffraction pattern was also taken from the interface of matrix and twin. By comparing the TEM and the diffraction images, the twin plane was determined. The twin direction was found by knowing that it lies on the twinned plane (i.e. the common plane) and perpendicular to the direction which brings the twin diffraction pattern coincident with that of the matrix.

In our case, the twins were of $\{1\bar{1}02\}\langle 10\bar{1}\bar{1}\rangle$ type.

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BOOK PAGE

TITLE TEM ANALSIS OF GR. 7 (cont.) PROJECT

Continued From Page

SPECIMEN: G00556

This sample id (G00556) was deformed at 150°C at 85% Y.S.

The TEM showed the presence of a-type screw dislocation ($\bar{b} = \frac{1}{3}\langle 11\bar{2}0 \rangle$, slip plane $\{1\bar{1}00\}$).

There was no sign of deformation twinning.

SAMPLE ID: F37112

This sample was deformed at 100°C at 85% Y.S.

The TEM ~~m~~ results showed the presence of a-type screw dislocations predominantly with no indication of twinning.

The results were similar to the one obtained at 150°C at 85% Y.S.

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SIGNATURE [signature] DATE 01/11/08

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~~PROPRIETARY INFORMATION~~ 6/22/10 [initials]

TITLE TEM sample preparation PROJECT

Continued From Page

GRADE 7

TEM samples of Grade 7 were prepared using the method described on pages 2 and 3.

The samples were: G01025 and G00563 (deformed at 85% Y.S. and 200°C and 250°C, respectively); F37105, F37113 and F37121 (deformed at 70% Y.S. and 25°C, 100°C and ~~F3~~ 250°C).

Seven (7) TEM samples were made from each specimen for future TEM analysis.

GRADE 5

For the first time, specimens of Grade 5 were cut and TEM samples were made similar to that described for Grade 7 alloy.

The main difference in sample preparation lied in the chemical used for twin-jet polishing. Here, a solution of 84% methanol, 10% butanol and 6% perchloric acid was used and the polishing was carried out at 10V.

The samples that were cut into TEM specimens were: G00572, G00573, G00574, G00568, G01028 and G00575 (all deformed at 85% Y.S. and at temperatures of 25°C, 50°C, 100°C, 150°C, 200°C and 250°C, respectively).

Continued To Page

SIGNATURE [signature] DATE 01/18/2008

DISCLOSED TO AND UNDERSTOOD BY DATE

~~PROPRIETARY INFORMATION~~ 6/22/10

TITLE CREEP CURVES ANALYSIS **PROJECT**

Continued From Page

GRADE 7

Creep data of Grade 7 alloy obtained during Phase II were plotted using MS Excel and were fitted with the known creep equations, $\varepsilon = A' + B \ln t$ and $\varepsilon = At^n$. The study was done on all the creep curves deformed at 85% Y.S. at different temperatures and at 150°c at different ~~stree~~ stresses.

At lower stress levels such as 40% Y.S. and 55% Y.S., $\varepsilon = At^n$ seemed a better fit.

At higher stress levels, however, the log curve gave a better fit to the experimental curve.

From the creep curves at 150°c at different stresses, a threshold stress level was determined at which creep is negligible. The calculation gave ($\varepsilon_{min.} = 0.02\%$) a threshold stress value of 34.5% Y.S.

Activation energy of the creep process ~~co~~ was also computed at 85% Y.S.

It was observed that the creep curves didnot necessarily increase with increase in temperature. It initially increased, reaching a maximum strain at 100°c, and then decreased monotonically with temperature. Such a phenomenon was also observed during Phase I.

It was important to calculate the activation energies at different strains. The activation energy (Q) was computed using the following equation

$$Q = -R\left[\frac{\Delta \ln \dot{\varepsilon}}{\Delta(1/T)}\right]$$; where R = gas constant = 8.31 J/k mol

T = Temperature in Kelvin

$\dot{\varepsilon}$ = strain rate

The table below shows Q as a function of strain (in percent)

Strain (%)	Q KJ/mol
0.1	27.1
0.2	27.0
0.4	22.9
1.0	~~41.5~~ 53.6 AB
1.5	63.3
2.0	~~53.6~~ 61.5 AB

Continued To Page

SIGNATURE [signature] DATE 01/21/2008

DISCLOSED TO AND UNDERSTOOD BY DATE

~~PROPRIETARY INFORMATION~~ 6/22/10 MP

TITLE CREEP CURVES ANALYSIS **PROJECT**

Continued From Page

GRADE 5

Creep curves of Grade 5 alloy were plotted and the best fit equation was determined, at 85% Y.S. at different temp-eratures and at 150°C at different stresses.

It was noticed that for the Phase II experimental data, the power-law equation, $\varepsilon = At^n$ was a better fit at all the different temperatures and stresses.

Threshold stress value for Grade 5 was obtained at 150°C ($\varepsilon_{min} = 0.02\%$) by plotting the stress as a function of creep strain. The value was found to be 44% Y.S.

Activation energies were similarly computed at 85% Y.S. The creep curves, similarly, did not increase monotonically with temperature.

The activation energies were found not to vary dramatically with strain. as was the case for Grade 7 alloy.

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DATE 01/23/2008

DISCLOSED TO AND UNDERSTOOD BY

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~~PROPRIETARY INFORMATION~~ 6/22/10

TITLE TRANSMISSION ELECTRON MICROSOPY. **PROJECT**

Continued From Page

GRADE 7

TEM analyis is performed on G01025 and ~~G0to~~ G00563. Both the specimens showed a-type screw dislocations with no deformation twinning.

GRADE 5

TEM analysis is done for all the samples deformed at 85% Y.S. at different temperatures.

The Grade 5 alloy is a mixture of two-phases i.e. α & β. α is hcp whereas β is bcc. The TEM picture reveals these two different phases by β-phase appearing as a lamellar ~~stun~~ structure. Additional verification is done by taking the diffraction pattern from α-phase & β-phase.

A uniform observation is that most of the deformation is happening in α-phase through process of dislocation motion whereas the β-phase appears to be dislocation free.

The sample IDs are: G00572, G00573, G00574, G00568, G01028 and G00575.

Continued To Page

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DATE 02/26/2008

DISCLOSED TO AND UNDERSTOOD BY

DATE

~~PROPRIETARY INFORMATION~~ 6/22/10

TITLE TEM SAMPLE PREPARATION **PROJECT**

Continued From Page

GRADE 5

TEM samples of Grade 5 alloy deformed at 70% Y.S. at different temperatures are ~~cut~~ prepared as described on pg. 11.

Sample Id : E65387 ; E65395 ; E65411

These samples are to be studied during Phase II, but were received earlier (before the start of Phase II).

Continued To Page

SIGNATURE [signature] DATE 03/19/2008

DISCLOSED TO AND UNDERSTOOD BY DATE

~~PROPRIETARY INFORMATION~~ 6/22/10MP

TITLE Transmission Electron Microscopy **PROJECT**

Continued From Page

GRADE 7

TEM analysis is done on F37105 (70% at 25°c).

The sample showed a-type screw dislocations with no presence of deformation twinning.

Similar results were also obtained for F37113 (70% at 100°c) and F37121 (70% at 250°c).

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TITLE Transmission Electron Microscopy **PROJECT**

Continued From Page

Grade 7

TEM analysis of F37103 (100% Y.S. at 25°C)

Seven TEM samples are prepared.

Most of the samples were found to be good for carrying out TEM examinations.

The analysis showed a-type screw dislocations. The glide plane was prism $\{1\bar{1}00\}$ plane. Additionally, twinning was also seen in most of the samples.

The twinning type was found to be of $\{1\bar{1}02\}$ twin.

Continued To Page

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DATE 05/29/08

DISCLOSED TO AND UNDERSTOOD BY

DATE

TITLE Transmission Electron Microscopy **PROJECT**

Continued From Page

Grade 7

Finally, TEM analysis was carried out for sample id ~~F37~~ F37119 (100% Y.S. at 250°C).

The results showed slip and twinning present in the sample.

Continued To Page

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DATE 06/16/08

DISCLOSED TO AND UNDERSTOOD BY

DATE

~~PROPRIETARY INFORMATION~~ 6/22/10

TITLE Tensile & Creep Data Analysis **PROJECT**

Continued From Page

(1) Titanium Grade 7 alloy creep data at 25°C, 100°C & 250°C were plotted using Microsoft Excel and the analysis of the creep curves were carried out similar to the ones at 150°C.

The analysis involved obtaining the best fit logarithmic equation $\varepsilon = A' + B \ln t$, and the power law equation $\varepsilon = At^n$ at a given stress level and temperature.

For 25°C, A′ and B were then plotted as a function of the stress level and thereby ε was obtained as a function of the stress.

It was found that

$$\varepsilon = -0.013\sigma + 0.6722 + 3.11 \times 10^{-5} e^{+0.109\sigma} \ln(t).$$

The files were stored on the computer under each individual folder. For ~~ex~~ example, a folder "Grade 7" was created in which additional folders such as "25 degrees" etc. were created. Within "25 degrees" folder, there were individual excel data charts corresponding to each stress level. The files were named as 40 percent, 55 percent and so on, to indicate that the data belonged to 40 percent yield stress at 25 degrees C etc.

Such files were created for each temperature (i.e. 25°C, 100°C, 200°C). The data belonging to the file "Rest" belonged to the study done during the stage I final report and included the excel charts belonging to 150°C and 85 percent yield stress. The file also contained the tensile plots.

(2) Titanium Grade 5 analysis was similarly followed to that of Grade 7.

Continued To Page

SIGNATURE [signature] DATE 06/27/08

DISCLOSED TO AND UNDERSTOOD BY DATE

~~PROPRIETARY INFORMATION~~ 6/22/10

TITLE CREEP DATA ANALYSIS **PROJECT**

Continued From Page

GRADE 7.

1. Creep Analysis at 25°C

Based on the best fit logarithmic and power law equation with the expt. data, the logarithmic equation was selected for predicting creep deformation strain after a long period of time (more than 10hrs).

The basis for selecting log equation was mainly due to the observation of deformation twinning in samples deformed to large strains. Twinning is known to restrict creep deformation due to sub-division of the grains. Smaller grains result in lower dislocation & twinning activity. This further results in lower creep deformation. A log. equation predicts lower creep strain compared to power law eqn. and therefore is more suitable in predicting deformation at large strains. Further, the samples with limited creep strain at lower stress levels would experience twinning activity as the strain increases with time, and hence log eqn. might be valid for such deformation condition after a very long period of time (around 10,000 yrs).

Additionally, log eqn. is able to predict the creep strain (based on the experimental data) from 10 hours to 200 hrs. using the eqn. derived by matching the experimental data from 0 to 10 hrs.

The time dependent creep constants for the power law eqn. A and n do not follow a particular trend with stress, thereby indicating a possibility of lower creep at higher strains. The constant B of the log eqn. exponentially increases with stress and therefore not indicating such a physically impossible situation.

2. creep analysis at 100°C, 150°C and 250°C follow similar trend to that at 25°C → universal use of log eqn. for predicting creep deformation.

Continued To Page

SIGNATURE [signature] DATE 06/28/08

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TITLE *CREEP DATA ANALYSIS.* PROJECT

Continued From Page

GRADE 5.

1. Creep analysis at 25°C.

Logarithmic eqn. is again selected over the power law eqn. based on the transmission electron microscopy results.

The results of transmission electron microscopy results indicate deformation restricted only in the α-phase at most of the stress levels (in fact, all at 25°c). The β-phase remains elastic during the creep deformation. Thus the dislocation motion is restricted at the α/β interphase boundary leading to lower creep deformation.

Since log eqn. predicts a lower creep strain compared to the power law eqn., the log eqn. is selected.

2. Similarly, log eqn is selected for creep deformation at 100°c, 150°c and 250°c.

Continued To Page

SIGNATURE [signature] DATE 03/28/08

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TITLE CREEP DATA ANALYSIS. PROJECT

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1. Threshold Stress

Threshold stress is defined as the limiting stress or the minimum stress below which there is undetectable creep strain.

a. Grade 7.

Temperature	Threshold Stress (MPa)	Threshold stress (Percent of Yield Stress)
25°C	148.0	40.7
100°C	55.2	19.8
150°C	65.5	34.5
250°C	52.5	31.5

As temperature increases, the threshold stress reaches a minimum at 100°C due to the highest drop in flow stress above 100°C causing the threshold stress to increase.

b. Grade 5

Temperature	Threshold Stress (MPa)	Threshold Stress (Percent of Yield Stress)
25°C	484.8	48.0
100°C	410.8	46.0
150°C	367.8	44.0
250°C	235.2	31.5

As temperature increases, expectedly, the threshold stress decreases.

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DATE 02/30/08

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~~PROPRIETARY INFORMATION~~ 6/22/10

TITLE CREEP DATA ANALYSIS **PROJECT**

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2. Failure stress

Failure stress is defined as the stress for which the creep strain strain equals the strain at ultimate tensile strength, where instability in tension occurs, after a period of time. The time period selected in the present study is 10,000 years.

a. Grade 7

Temperature	Failure Stress (MPa)	Failure Stress (Percent Yield Stress)
25 °C	326.3	89.7
100 °C	226.9	81.4
150 °C	197.2	90.3
250 °C	> 166.7°	>100° (not expected to fail at 100 percent)

As temperature increases, the failure stress decreases. However, as percentage of yield stress, it has a minimum at 100 °C due to the drop in flow stress (the way it is computed).

b. Grade 5

Temperature	Failure Stress (MPa)	Failure Stress (Percent yield stress)
25 °C	> 1010	>100
100 °C	> 893	>100
150 °C	> 836	>100
250 °C	> 747	>100

The samples of Grade 5 are not expected to fail at these temperatures.

Continued To Page

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DATE 06/30/08

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~~PROPRIETARY INFORMATION~~ 6/22/10