

HIGH STRAIN RATE PROPERTIES OF TANTALUM PROCESSED BY EQUAL CHANNEL ANGULAR PRESSING

Philip J. Flater
Joel W. House
Air Force Research Laboratory
Munitions Directorate
AFRL/MNMW
Eglin AFB, FL 32542-6810

James M. O'Brien
O'Brien & Associates
406 S. Lane Street
Blissfield, MI 49228

William F. Hosford
University of Michigan
Materials Science & Engr.
2300 Hayward Street
Ann Arbor, MI 48109



AUGUST 2007

CONFERENCE PAPER AND BRIEFING CHARTS

The paper and corresponding briefing charts were presented at the 15th APS Topical Conference on Shock Compression of Condensed Matter, 24-29 June 2007, Kohala Coast, Hawai'i. Only the paper will be published by the American Physical Society in the unclassified/unlimited distribution proceedings. One or more of the authors are U.S. Government employees working within the scope of their position; therefore, the U.S. Government is joint owner of the work. If published, the American Physical Society may assert copyright. Any other form of use is subject to copyright restrictions.

This paper is published in the interest of the scientific and technical information exchange. Publication of this paper does not constitute approval or disapproval of the ideas or findings.

DISTRIBUTION A: Approved for public release; distribution unlimited.

Approval Confirmation

#AAC/PA 07-30-07-499 for the conference paper, dated 30 July 2007
and #AAC/PA 06-15-07-453 for the briefing charts, dated 15 June 2007.

AIR FORCE RESEARCH LABORATORY, MUNITIONS DIRECTORATE

REPORT DOCUMENTATION PAGE					<i>Form Approved OMB No. 0704-0188</i>	
The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing the burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.						
PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.						
1. REPORT DATE (DD-MM-YYYY)		2. REPORT TYPE			3. DATES COVERED (From - To)	
4. TITLE AND SUBTITLE				5a. CONTRACT NUMBER		
				5b. GRANT NUMBER		
				5c. PROGRAM ELEMENT NUMBER		
6. AUTHOR(S)				5d. PROJECT NUMBER		
				5e. TASK NUMBER		
				5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)					8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)					10. SPONSOR/MONITOR'S ACRONYM(S)	
					11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT						
13. SUPPLEMENTARY NOTES						
14. ABSTRACT						
15. SUBJECT TERMS						
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON	
a. REPORT	b. ABSTRACT	c. THIS PAGE			19b. TELEPHONE NUMBER (Include area code)	

HIGH STRAIN-RATE PROPERTIES OF TANTALUM PROCESSED BY EQUAL CHANNEL ANGULAR PRESSING

P. J. Flater¹, J. W. House¹, J. M. O'Brien², and W. F. Hosford³

¹*Air Force Research Laboratory/Munitions Directorate, 101 W. Eglin Blvd, Eglin AFB, FL 32542*

²*O'Brien & Associates, 406 S. Lane St, Blissfield, MI 49228*

³*University of Michigan, Materials Science & Engr, 2300 Hayward St, Ann Arbor, MI 48109*

Abstract. Current ingot refinement and solidification techniques used in tantalum processing often result in inconsistent mechanical properties. Subsequent processing by equal channel angular pressing (ECAP) has been shown to reduce or eliminate internal structural variations as well as part-to-part variability [2]. This paper presents the effects of ECAP processing on the properties of tantalum. The materials of interest are 2.5-inch round bar tantalum supplied by H.C. Starck and Cabot Supermetals. Three metallurgical conditions were examined for each material: as worked, fine-grain annealed, and large-grain annealed. Prior to annealing, each bar was processed eight times through a 135-degree ECAP die using route C and then forged into 0.25-inch thick plates. Mechanical property specimens were subsequently removed from the plates for low and high-rate uniaxial compression experiments. Orientation dependence was characterized by orienting specimen load axes through the thickness or in the plane of the forged plate. Wave propagation and anisotropy were studied using Taylor impact experiments.

Keywords: Tantalum, Taylor impact, ECAP, ECAE

PACS: 62.20.-x, 62.20.Fe, 62.50.+p

INTRODUCTION

Developing forged plate tantalum with consistent mechanical and physical properties is an area of interest for high strain-rate applications. Mill practices for pure tantalum, specifically ingot refinement and solidification, often lead to inconsistent mechanical properties within the processed material. Evidence of the mill practices often continues through subsequent thermo-mechanical processing steps even with high levels of induced plastic work or recrystallization. This heterogeneity has been observed in upset forged tantalum plates where the processing history manifests in the form of texture banding [1]. Distinct regions of hard (111) and soft (100)

crystallographic regions are encountered as one passes through the thickness of a forged plate (Figure 1). Research has shown that processing tantalum by equal channel angular pressing (ECAP) can reduce microstructural heterogeneities [2]. Thus it is of interest to examine the textural and microstructure modification by ECAP processing on tantalum plate material. The objectives of this research are to (1) process pure tantalum by ECAPing, forging, and annealing and (2) characterize the effects of the thermo-mechanical processing on the mechanical properties. The ultimate goal is to generate consistent structural properties of tantalum for high strain-rate applications.

Material

The materials of interest are commercially pure tantalum from HC Starck and Cabot Supermetals. All material was mechanically processed by ECAP eight passes using route C. The material was then forged into 0.25-inch thick plates. Three metallurgical conditions were investigated for each supplier's material: as worked (ECAP + forging only); annealed for 1 hour at 950°C (Cabot) or 1050°C (Starck) to produce an average grain diameter of 20 μm (fine grain); and annealed for 1 hour at 1250°C to produce an average grain diameter of 100 μm (large grain). Specimens were removed from the through-thickness (TT) and radial in-plane (IP) orientations of the forged plates, and annealing was performed within a vacuum furnace.

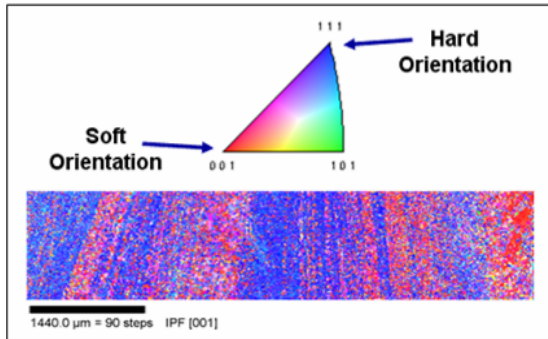


Figure 1. Texture banding through the thickness (left to right) of forged tantalum plate. [1]

Mechanical properties experiments

A series of quasi-static, split-Hopkinson pressure bar, and Taylor impact experiments as well as microhardness measurements were performed to characterize the materials in all three metallurgical conditions. The orientation dependencies of each metallurgical condition were analyzed relative to the IP and TT directions.

Taylor impact experiments were performed on specimens removed in the plane of the plates to characterize the dynamic material response. The Taylor impact experiment involves a right circular cylinder specimen (diameter = 5.33 mm, L/D = 10) launched into a rigid surface [3]. High-speed photography captures the dynamic deformation

event while the impacted specimen can be recovered for postmortem analysis.

RESULTS AND DISCUSSION

An annealing study was performed to characterize the recrystallization behavior of the Cabot and Starck materials. Specimens were removed from the ECAPed and forged plates and annealed for one hour. Hardness measurements were made on the annealed samples, and the data is given in Figure 2. The hardness data indicates that the Cabot material recrystallizes at $\sim 125^\circ\text{C}$ lower temperature than the Starck material. Comparison of the data for samples oriented in the IP or TT direction revealed little orientation dependence, except in the as worked condition where the TT direction had higher values for both mills.

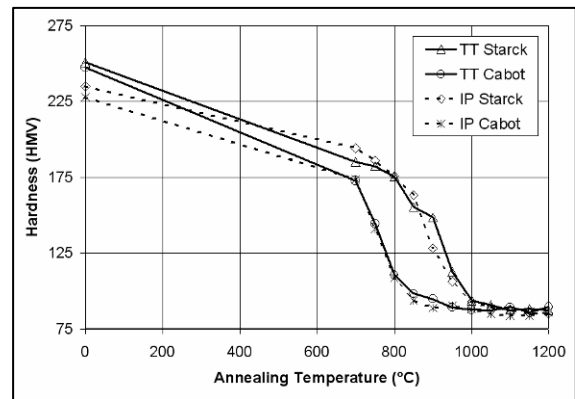


Figure 2. Vickers microhardness versus annealing temperature for ECAP and forged Cabot and Starck materials.

Low rate compression

Quasi-static compression experiments were performed at a strain rate of 0.01 s^{-1} for all three metallurgical conditions: as worked (AW), fine-grain anneal (FG), and large-grain anneal (LG). Data for specimens loaded in the IP direction are given in Figure 3 and for specimens loaded in the TT directions in Figure 4.

The mechanical properties at low rates show some level of orientation dependence, but no appreciable grain size effect. For both mills, the TT direction gave the highest yield strength values. In

the as worked condition, Cabot had slightly higher yield strength than Starck. It was noted that in the TT direction the as worked material still had some strain hardenability. While the hardening rates are linear, for the as worked TT direction, Starck had a slightly higher rate of hardening. In contrast, neither as worked material hardened in the IP direction. The as worked mechanical properties ranged from 800-900 MPa for the forged plate. Mechanical properties of these magnitudes have been attributed to tantalum wire with diameters of 1 mm or less [4].

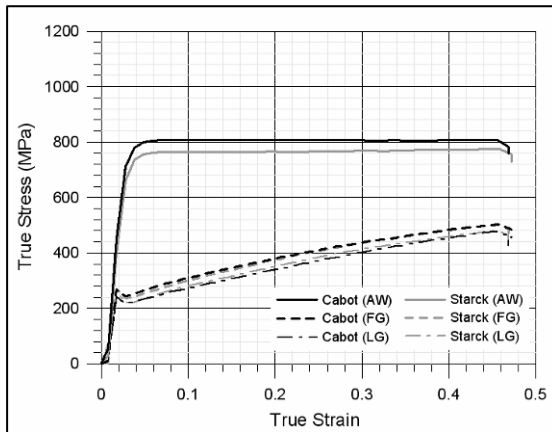


Figure 3. Low rate mechanical property data for Cabot and Starck (IP) specimens.

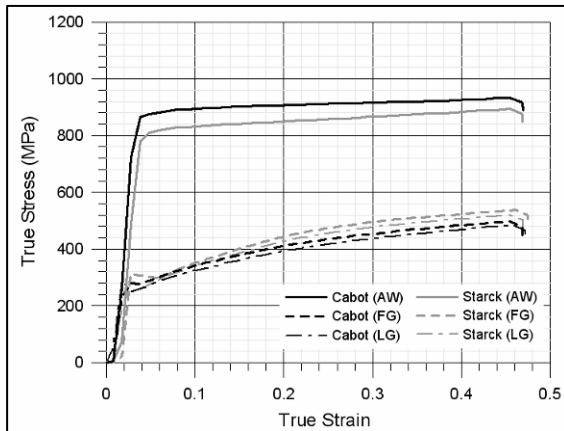


Figure 4. Low rate mechanical property data for Cabot and Starck (TT) specimens.

The annealed materials (fine and large grain) oriented in the IP direction exhibit linear hardening, and the data in Figure 3 shows little

difference in mechanical properties between the two mills. For strains greater than 0.1, the stress levels are visually indistinguishable. In the TT direction, both materials have a slight non-linear hardening rate. Again, the Starck material has the higher hardening rate of the two.

High rate compression

High rate compression experiments were performed using a split-Hopkinson pressure bar (SHPB) with the results shown in Figures 5 and 6. Like the low rate experiments, the TT direction exhibits higher yield strengths than the IP direction. For the as worked condition in the IP direction, the stress level saturates at a true strain of 0.06. Above a true strain of 0.06, the stress decreases. In the TT direction, the as worked materials have 1 GPa yield strengths, with little subsequent ability to strain harden. Above a true strain of 0.1, the stress level decreases with increase strain.

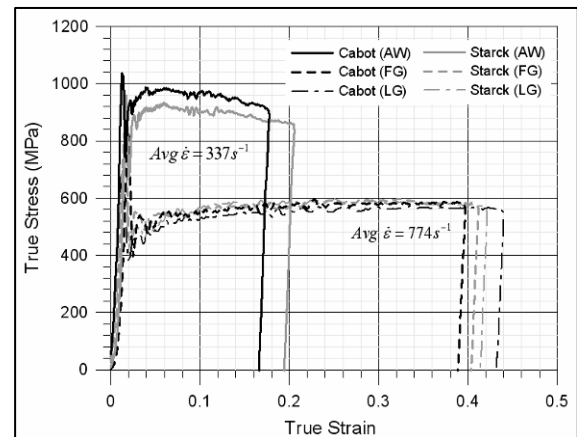


Figure 5. High rate mechanical property data for Cabot and Starck (IP) specimens.

The annealed materials in the IP direction have yield strengths at 440 MPa for both mills. These materials harden to a stress level of 580 MPa at a true strain of 0.25 to 0.35. In the TT direction, the annealed materials have average yield strengths of 560 MPa and harden to 610 MPa at a true strain of 0.1. Above a true strain 0.15, the stress decreases with increasing strain.

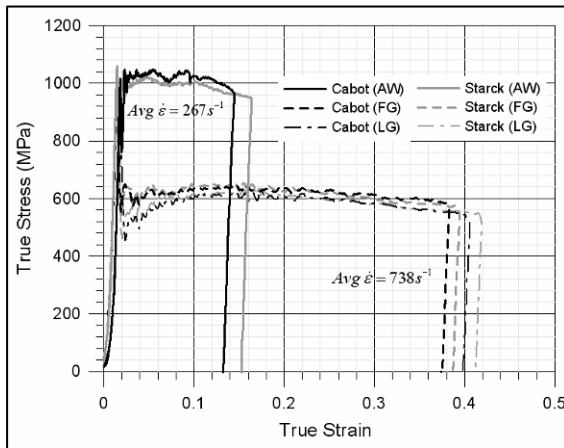


Figure 6. High rate mechanical property data for Cabot and Starck (TT) specimens.

Taylor impact

Taylor impact specimens were launched at an average impact velocity of 170 m/s. Analysis of the recovered specimen geometry showed strong mechanical anisotropy in the as worked and large grain materials, see Figure 7a. The extent of mechanical anisotropy was reduced in the fine grain material. In contrast, historical data for Cabot and Starck materials that had been forged and annealed (no ECAP) deformed in an isotropic manner (axisymmetric) upon impact, see Figure 7b. It should be noted that the current experiments (Figure 7a) are .21-caliber ECAP specimens with a length-to-diameter ratio of 10, and the historical experiments (Figure 7b) are .30-caliber forged specimens with a ratio of 5.

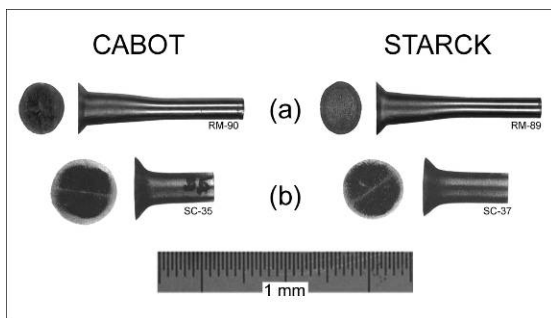


Figure 7. Profiles for recovered Taylor impact specimens processed by (a) ECAP and forging and (b) forging only. All specimens were annealed prior to testing.

CONCLUSIONS

Pure tantalum from Cabot Supermetals and H.C. Starck was processed by ECAP, followed by forging and annealing. Mechanical properties measured in the plane of the plate and through the thickness revealed an orientation dependence. In all cases, the through-thickness had higher yield strength values. Specimens recovered from Taylor impact experiments showed a distinct anisotropy to the deformation. This anisotropic response was most notable in the mushroom diameter which developed into an ellipse. Earlier work showed the deformation of forged and annealed material from the same suppliers to respond in an axisymmetric manner. The process of ECAP, forging, and annealing of tantalum from the two mills produced mechanical property data that was very consistent between the suppliers. The structures of the material that evolved through the thermo-mechanical processing generated a strong orientation dependence relative to the in-plane and through-thickness directions.

ACKNOWLEDGEMENTS

Portions of this research were funded under SBIR contract FO8651-02-C-0023. The authors acknowledge the support of the US Air Force Research Laboratory's, Munitions Directorate, and the contribution made by employees of InDyne Inc.

REFERENCES

1. House, J.W., Bingert, J.F., De Angelis, R.J., and O'Brien, J.M., "Thermo-Mechanical Processing of Forged Tantalum Analyzed by OIM and X-Ray Diffraction," Materials Science Forum, v 408-412 (I), 2002, pp 553-558.
2. Mathaudhu, S.N. and Hartwig, K.T., "Grain Refinement and Recrystallization of Heavily Worked Tantalum," MSE A, v 426, 2006, pp 128-142.
3. Taylor, G. I., "The use of flat-ended projectiles for determining dynamic yield stress. I: theoretical considerations," Proc. R. Soc. London. A, v 194, n 1038, 1948, pp 289-299.
4. "Tantalum / Niobium," Plansee SE, information brochure, 611 DE 10.06 (2000) RWF, p 13.

High Strain Rate Properties of Tantalum Processed by Equal Channel Angular Pressing



DISTRIBUTION A: Approved for public release; distribution unlimited.
Approval Confirmation #AAC/PA 06-15-07-453.

Philip J. Flater
Munitions Directorate
Air Force Research Laboratory

15th APS SCCM Conference, 24 - 29 Jun 2007



Acknowledgments



- **Co-authors**
 - Dr. Joel House
 - Dr. James O'Brien
 - Prof. William Hosford
- **Technicians**
 - Richard Harris, Harold Gilland, Tony Gomillion, Adam White
- **Students**
 - Brock Connolly, Beth Freisthler, Emily Ruff, David Williams



O'Brien & Associates
Metallurgical Consultants





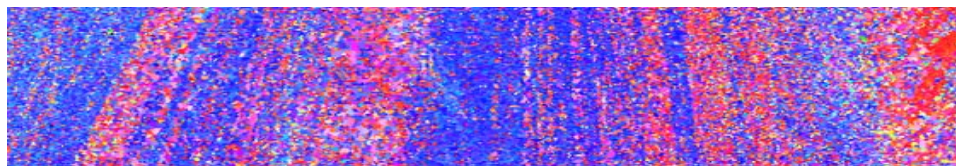
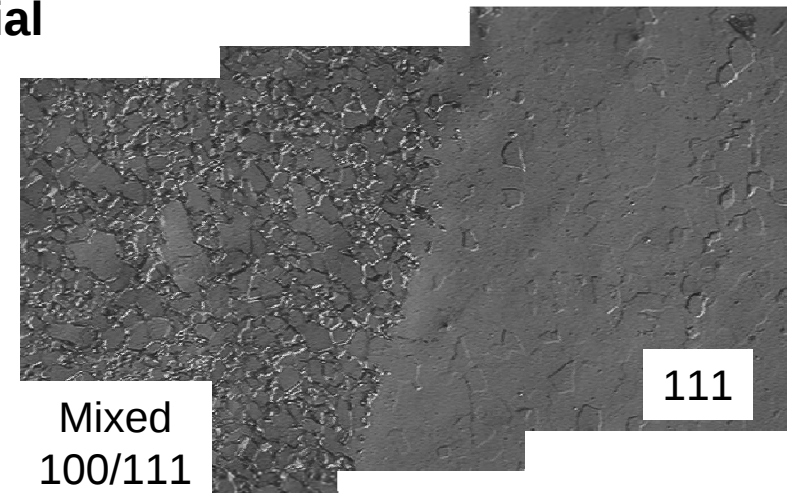
Outline



- **Material**
- **Objectives**
- **Physical Properties**
 - Optical microscopy
 - X-ray diffraction*
- **Mechanical Properties**
 - Quasi-static
 - High-rate (split Hopkinson pressure bar)
 - Taylor impact
- **Conclusions**

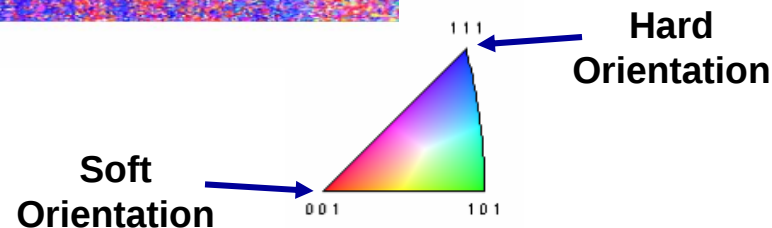


- Pure tantalum concerns
 - Lack of consistent mechanical properties due to mill practice
 - Ingot refinement
 - Solidification
 - Texture banding in forged plate material
 - Microstructural heterogeneities
- US suppliers
 - Cabot Supermetals
 - H.C. Starck



1440.0 μm = 90 steps IPF [001]

Source: John Bingert, LANL





Objectives



- **Objectives**
 - ECAP tantalum 8 passes route C
 - Forge material into 0.25"-thick plates
 - Characterize the effects of ECAP, forging, and annealing on mechanical properties
- **Long-term goal**
 - Generate consistent structural properties for high strain rate applications
- **Relevant studies**
 - Microstructural homogenization through equal channel angular pressing (ECAP) ^[1]
 - Uniform texture ^[2]
 - High rate properties of tantalum ^[3]



¹ Mathaudhu & Hartwig, MSE A 426, 2006

² House et al, ICOTOM, 2002

³ Chen, Gray, & Bingert, TMS, 1996



Material Characterization

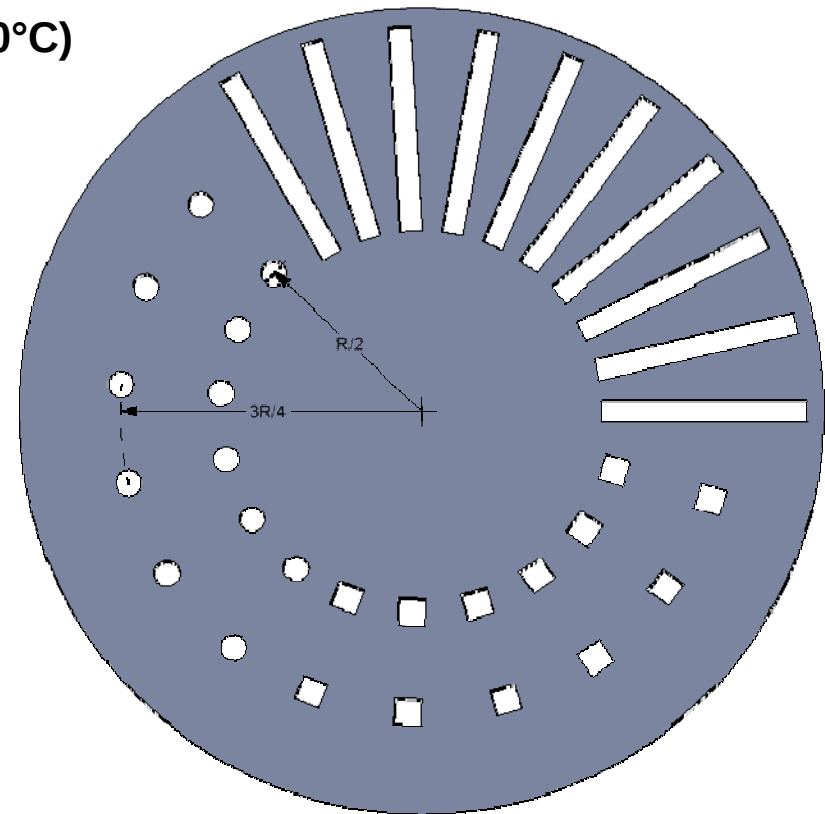


- **Baseline material**
 - **ECAP + Forge**
 - As worked
 - Fine grain (Cabot-950°C; Starck-1050°C)
 - Large grain (1250°C)
- **Mechanical property experiments**
 - **Compression**
 - In-plane & through-thickness
 - Quasi-static
 - High rate
 - **Taylor impact**
 - In-Plane

————— *Taylor Impact*

□ *IP Compression*

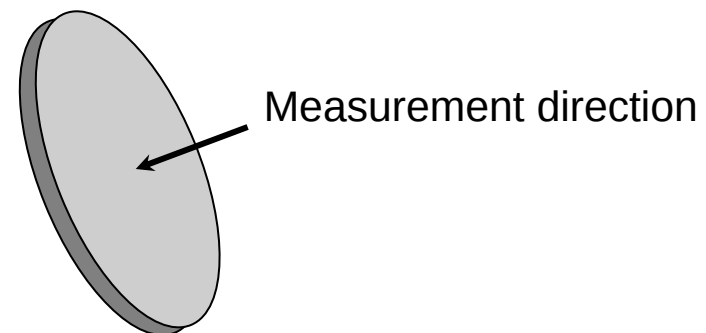
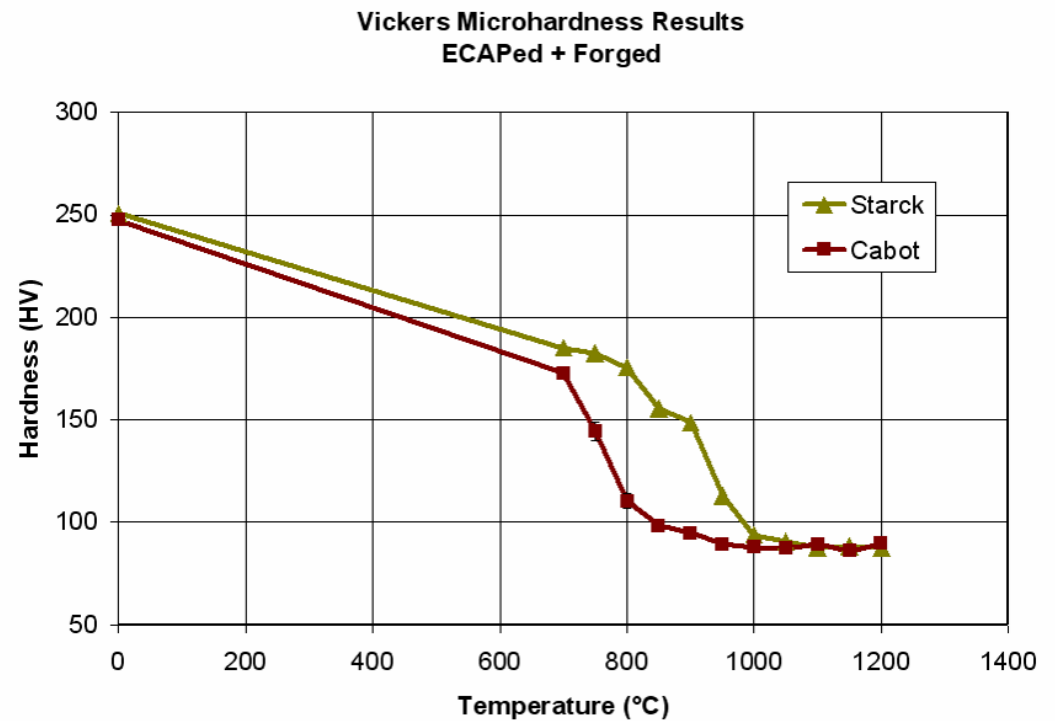
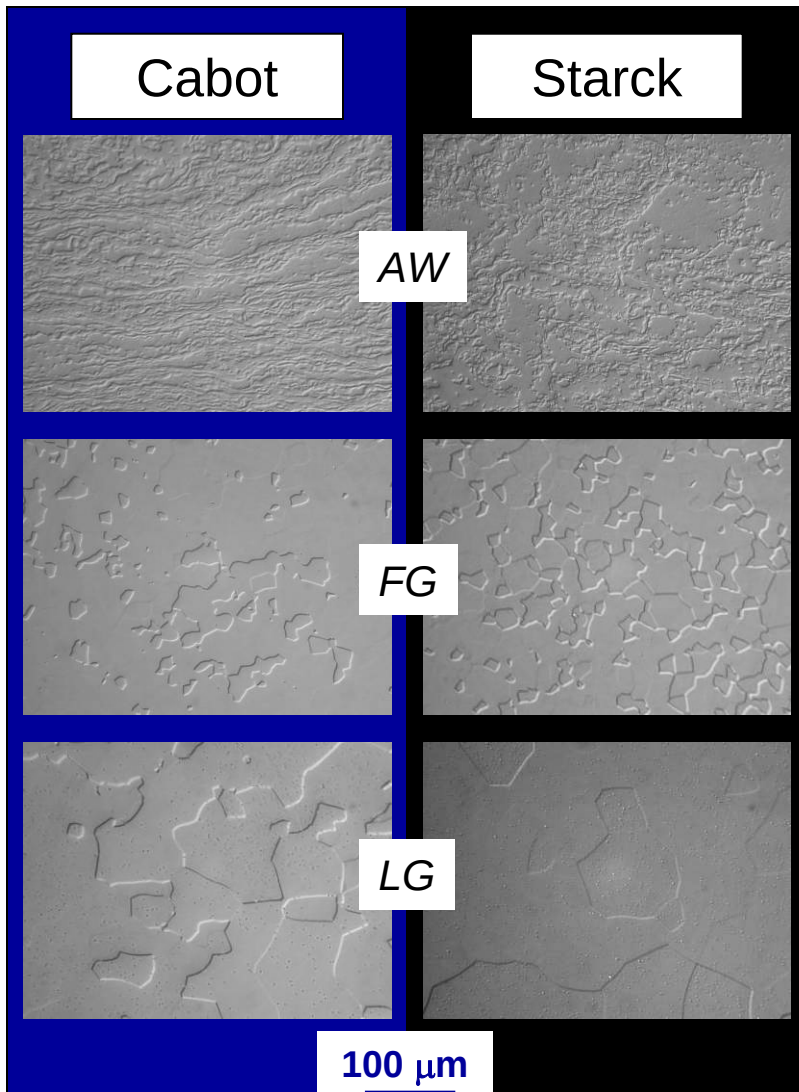
○ *TT Compression*





Annealing Study

Through thickness



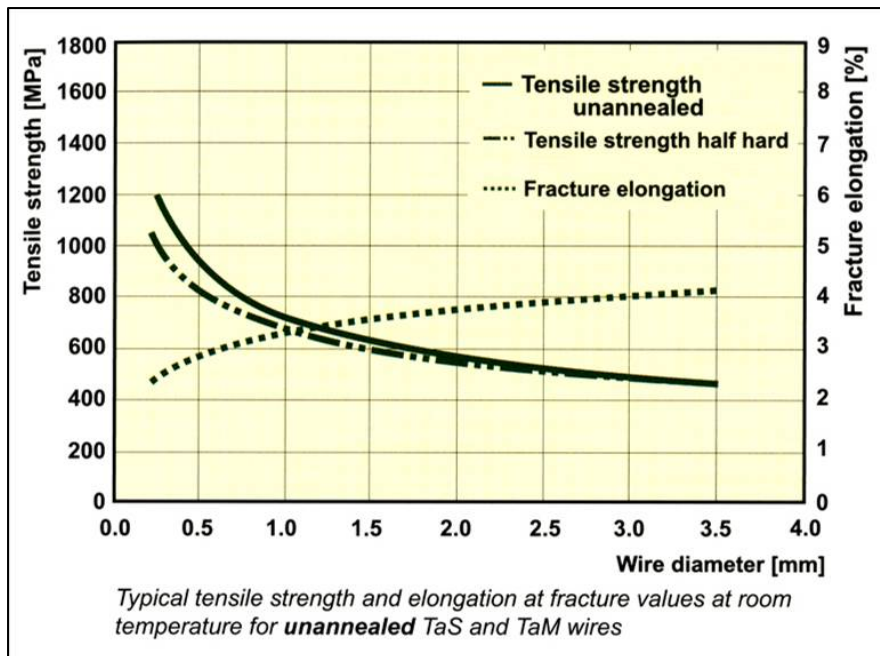


Mechanical Properties

Quasi-Static (0.01 s^{-1})

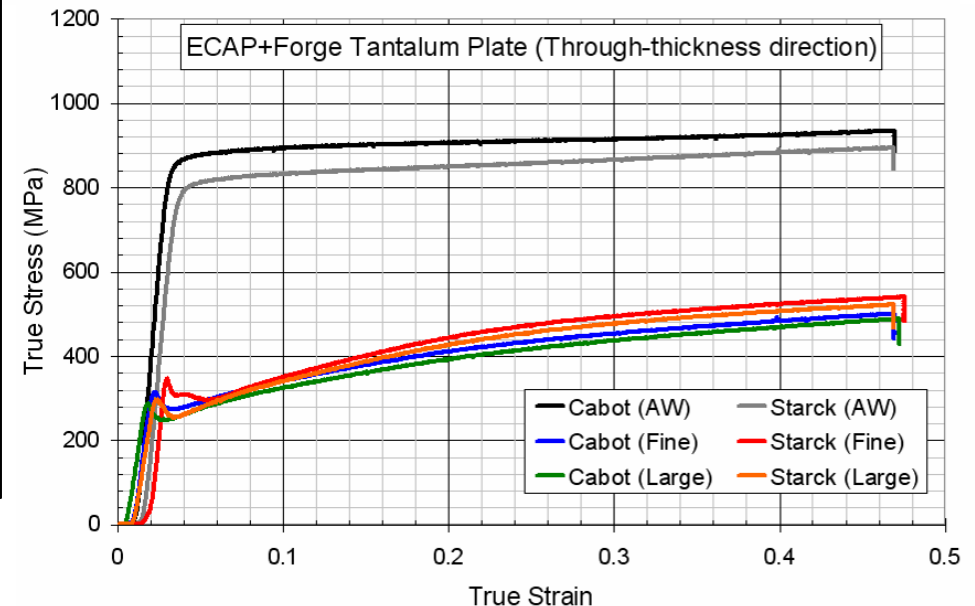
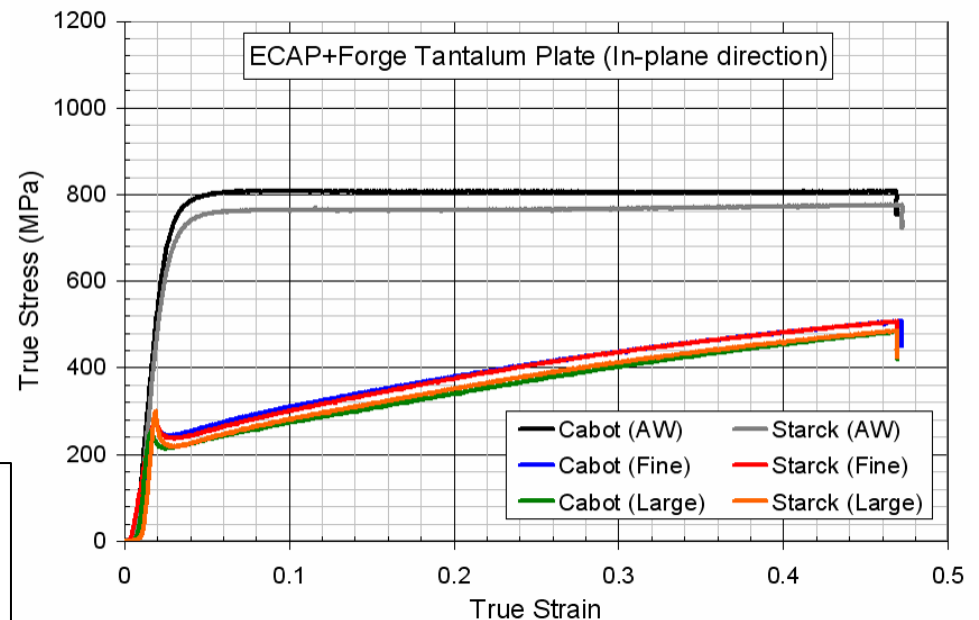


- Through thickness
 - Higher yield strength
 - Limited hardening
- In plane
 - No hardening



Source: Plansee

15th APS SCCM Conference , 24 - 29 Jun 2007



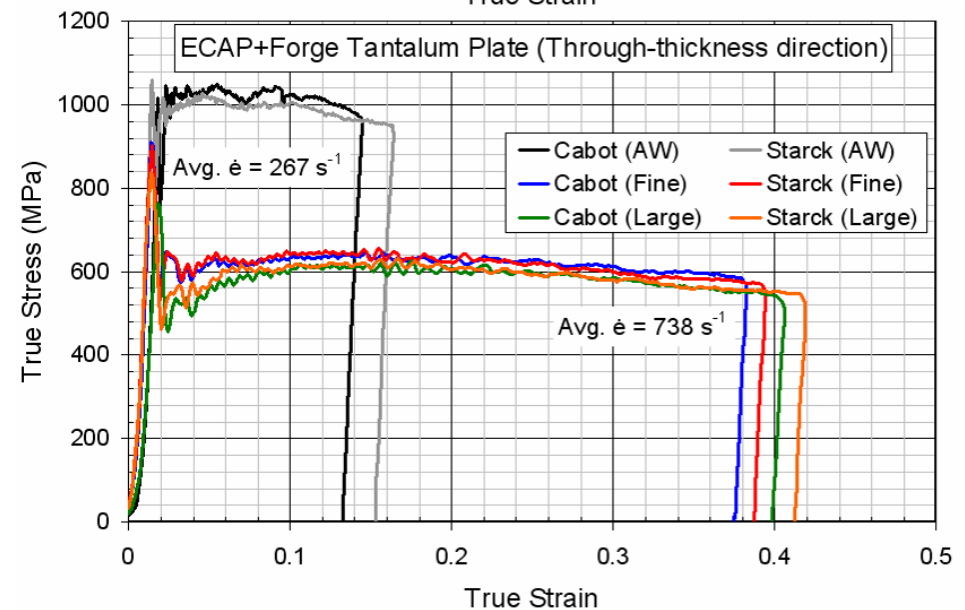
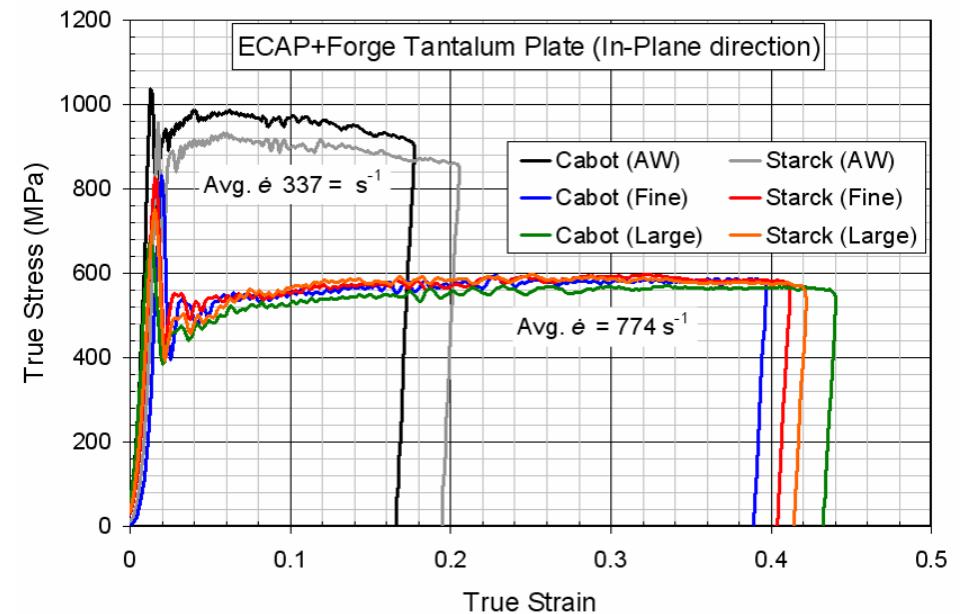
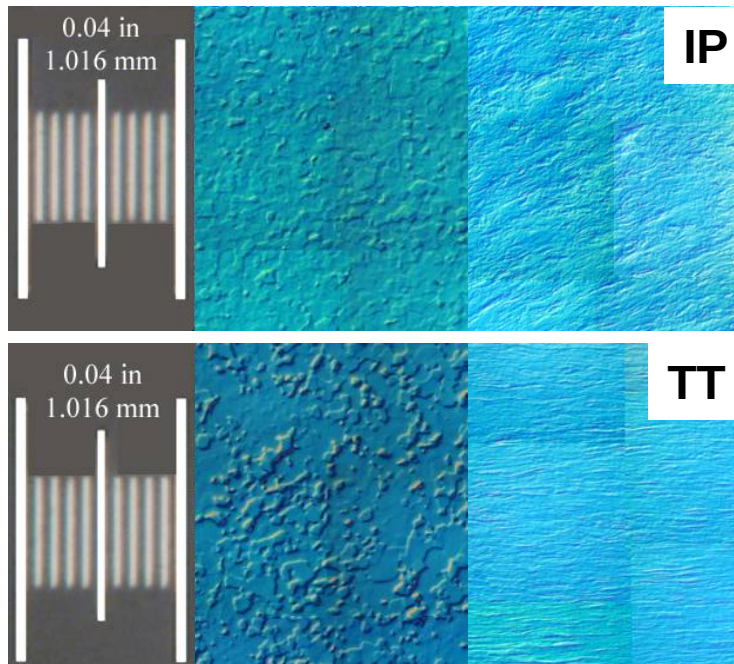


Mechanical Properties

Split-Hopkinson Pressure Bar



- Through thickness
 - Higher yield strength
- Limited thermal sensitivity
- Adiabatic condition
 - Consistent response between orientations





Impact Experiments



- **Taylor impact experiment (G.I. Taylor, 1948)**
 - **High-rate compression test**
 - **Compare profile of ECAP + forge material to forge only material**

Taylor Impact Experiment (RM89)

Tantalum (ECAP + Forge + Anneal)

Impact time = 132 μ s
Velocity = 169 m/s (554 fps)

P. Flater
H. Gilland
B. Connolly



Impact Experiments



Cabot



Avg Vel 170 m/s

AW

FG

LG

Starck





Conclusions



- **Mechanical property measurements were consistent between Cabot & Starck processed via ECAP**
- **Yield strength:**
 - Quasi-static ~850 MPa
 - High rate ~1000 MPa
 - Good agreement with the Plansee data for fine-diameter Ta wire
- **ECAP eliminated grain structure variations in plate material**
- **Subtle differences between IP & TT orientations**
- **Strong anisotropic material response in Taylor impact testing does not appear to be consistent with the limited orientation dependence found in the SHPB experiments**
- **FUTURE STUDIES – Detailed texture analysis of materials to characterize the extent of banding and texture evolution**