Bimodal Grain Distribution Control in Forged Inconel 751

PURDUE UNIVERSITY
School of Materials Engineering

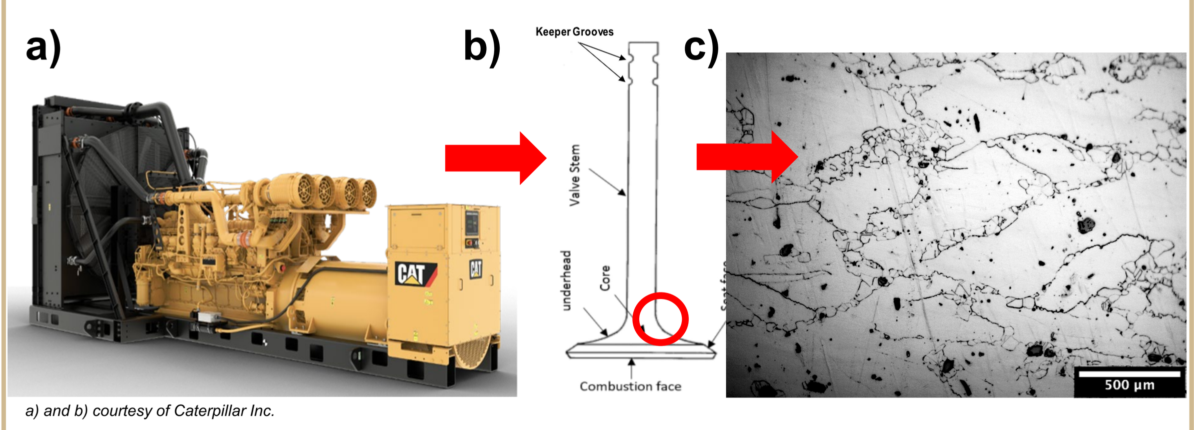
Student Names: Clay Barlow, Crystal Huang, Abbey Koneru, Akul Seshadri
Faculty Advisors: David Johnson
Industrial Sponsors: Caterpillar Inc.

Caterpillar has observed a bimodal grain distribution near the rounded edges of combustion valve heads used in a particular series of engines. The forged valves, comprised of Inconel 751, are vital to engine performance and the bimodal microstructure poses concerns for potential premature fatigue failure. This study aims to characterize the microstructure within the valve heads having undergone various processing and manufacturing conditions, and to develop a heat treatment plan that will minimize the occurrence of the bimodal grain size distribution. It was found that the microstructure within the valve heads differed based on initial forging conditions, but that a bimodal grain size was observed regardless. A post-forging heat treatment that includes high temperature solutionizing followed by two aging steps has been determined to best minimize the bimodal grain distribution, although further testing is needed to optimize heat treatment parameters.

This work is sponsored by Caterpillar Inc., Lafayette, IN

CATERPILLAR®

Project Background



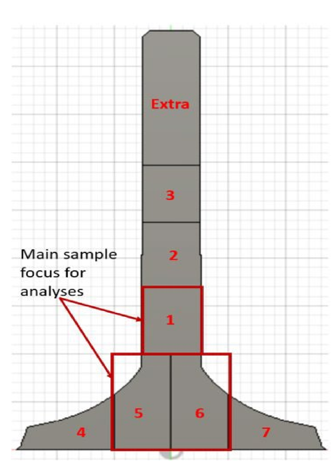
Problem: Bimodally distributed grains on the rounded edge that may affect the fatigue life of engine valves detrimentally

Experimental Procedure

Sample Preparation and Microstructure Characterization

Table 1: Valve sample case organizer

Case	Heat Treatment	Valve Supplier	Material Supplier	Forging Process
1	No heat treatment	A	A	A
2	Production heat treatment	A	A	A
3	No heat treatment	B	B	B
4	Production heat treatment	B	B	B
5	No heat treatment	B	C	B
6	Production heat treatment	B	C	B
7	Production heat treatment	B	B	B
8	Production heat treatment	B	C	B



- Cut-samples were mounted and polished up to 1 micron diamond paste and alumina slurry then etched using Marble's reagent
- Microstructures of samples characterized using optical microscopy and Scanning Electron Microscopy (SEM): Phenom Pro G6 Desktop SEM and a FEI Quanta 650 FESEM

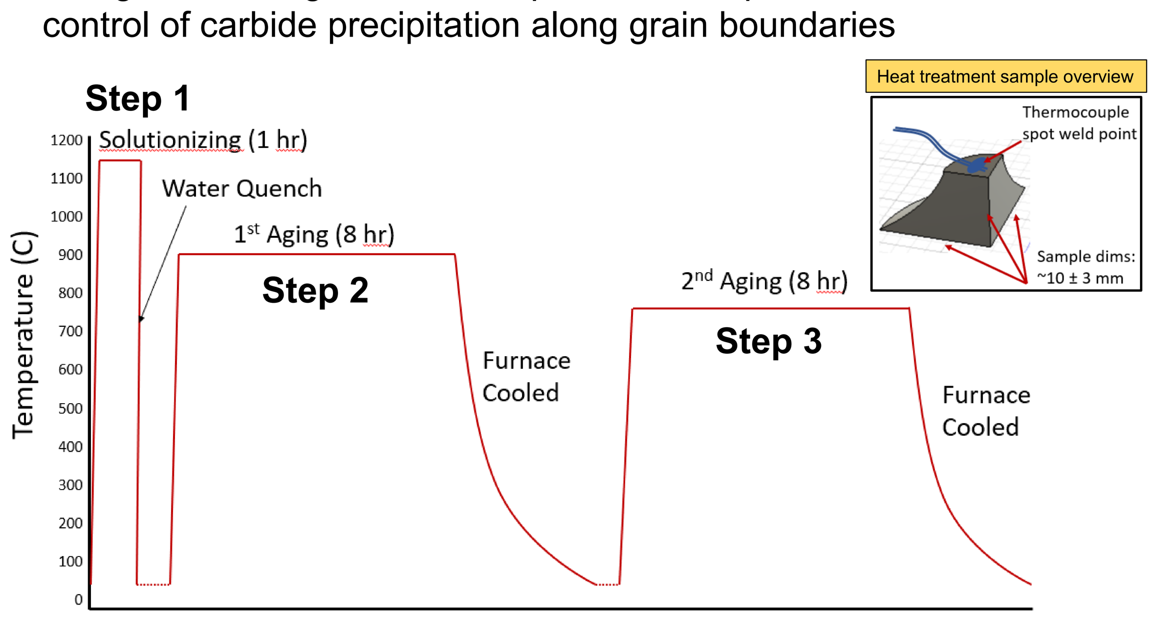
Heat Treatment Design

Motives to Design

1) An over occurrence of second phase carbides and a rapid growth of intermetallic phases (γ', γ'') are resulting in a bimodal grain distribution.

2) Solution annealing on forged material will dissolve second phase carbides and a double aging treatment will control carbide growth to minimize the occurrence of the bimodal grain size distribution by promoting more normalized grain growth.

- Designed utilizing Thermocalc phase development simulations for control of carbide precipitation along grain boundaries



Step 1 — Solutionizing (1140°C, 1hr)
- Dissolve precipitates and carbides, homogenize grains

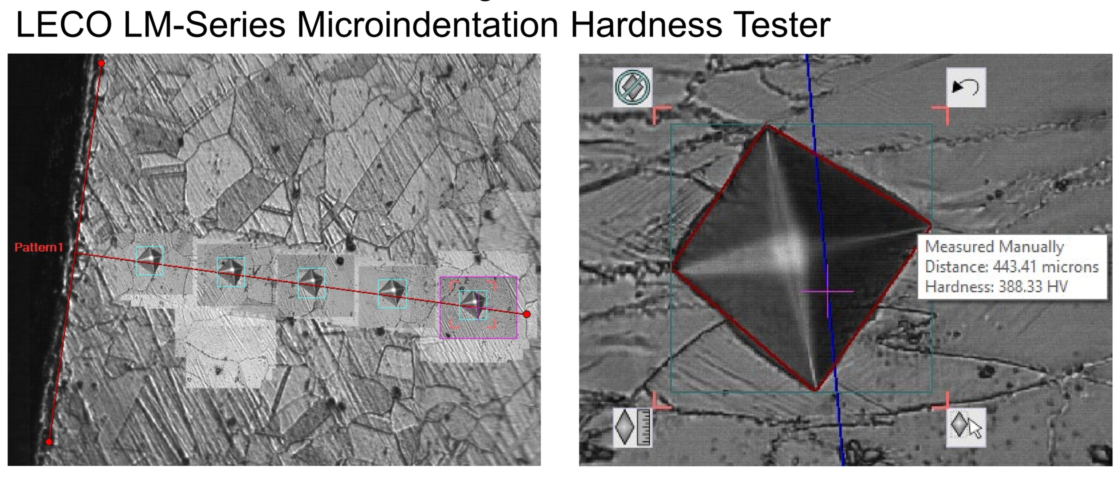
Step 2 — First Aging (900°C, 8hr)
- Allow rapid $M_{23}C_6$ and M_7C_3 carbide growth

Step 3 — Second Aging (750°C, 8hr)
- Encourage γ' growth for precipitation strengthening

- Carried out in a tubular furnace in air environment
- Type K thermocouple spot welded to sample for temperature control

Microindentation

- Mounted and polished samples were indented using a Vickers hardness indenter with a 500g load and 13 second dwell time on a LECO LM-Series Microindentation Hardness Tester

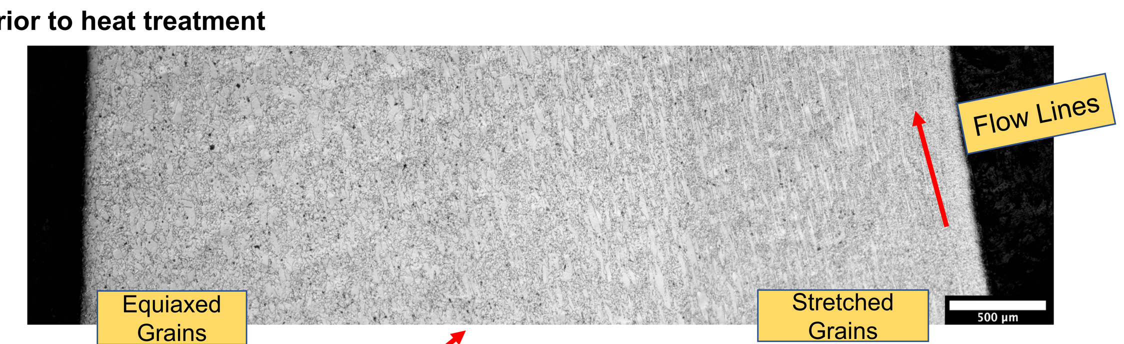


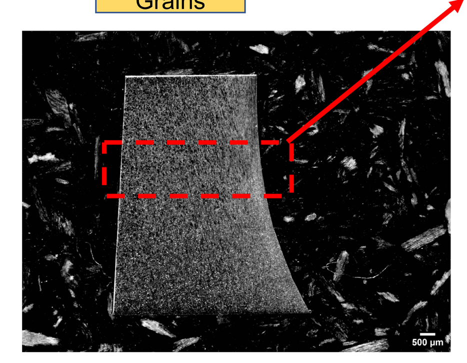
- Indentations were made in linear arrays from rounded edge and on selected areas of interest on the sample

Microstructure Evolution

Characterization of Purdue heat treatment valves

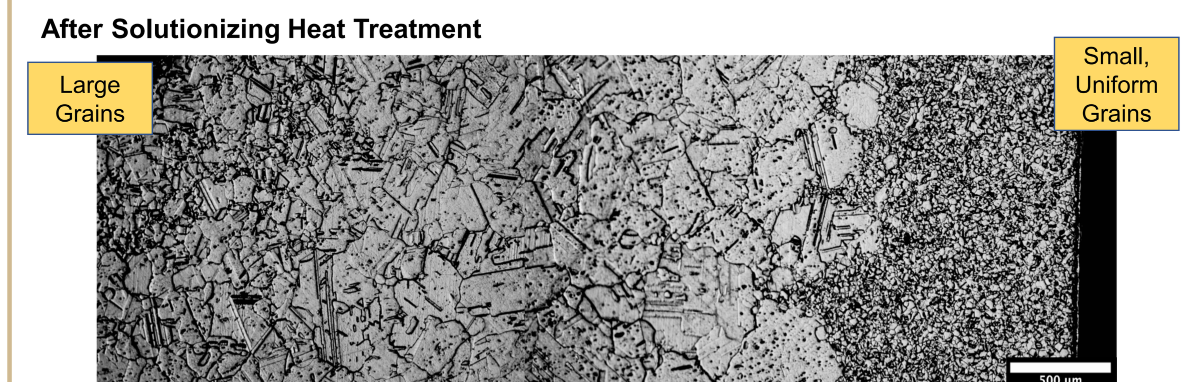
Prior to heat treatment

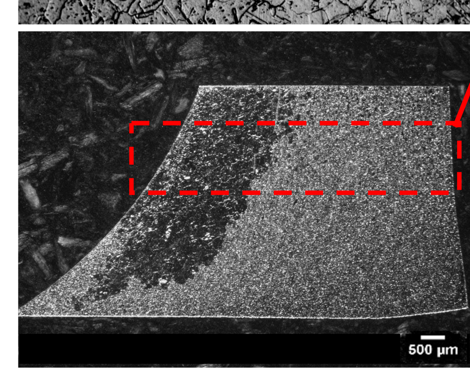




- Large, stretched grains intermixed with many small grains near the rounded edge, reflecting a bimodal grain distribution with post-forging flow lines
- Equiaxed grains near valve head center

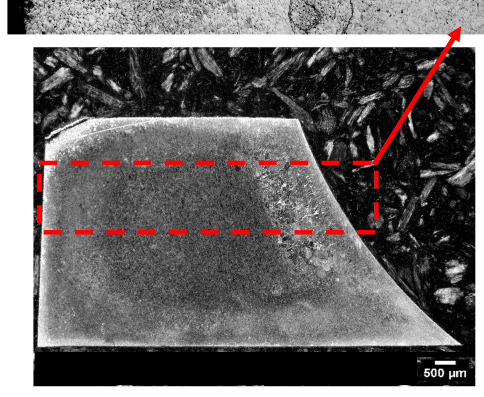
After Solutionizing Heat Treatment





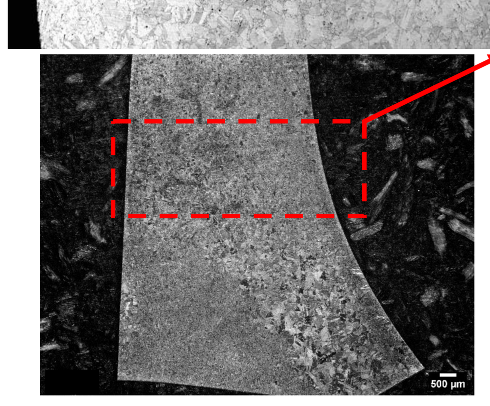
- Grain size and distribution is unexpected
- Bimodal grain sizes appear to be exacerbated
- Large concentrated area of large grains near the rounded edge
- Smaller more uniform grains everywhere else

After First Aging Treatment



- Bimodal distribution of grains is still present
- Concentrated area of large grains appears to decrease with overall grain distribution beginning to normalize

After Second Aging Treatment



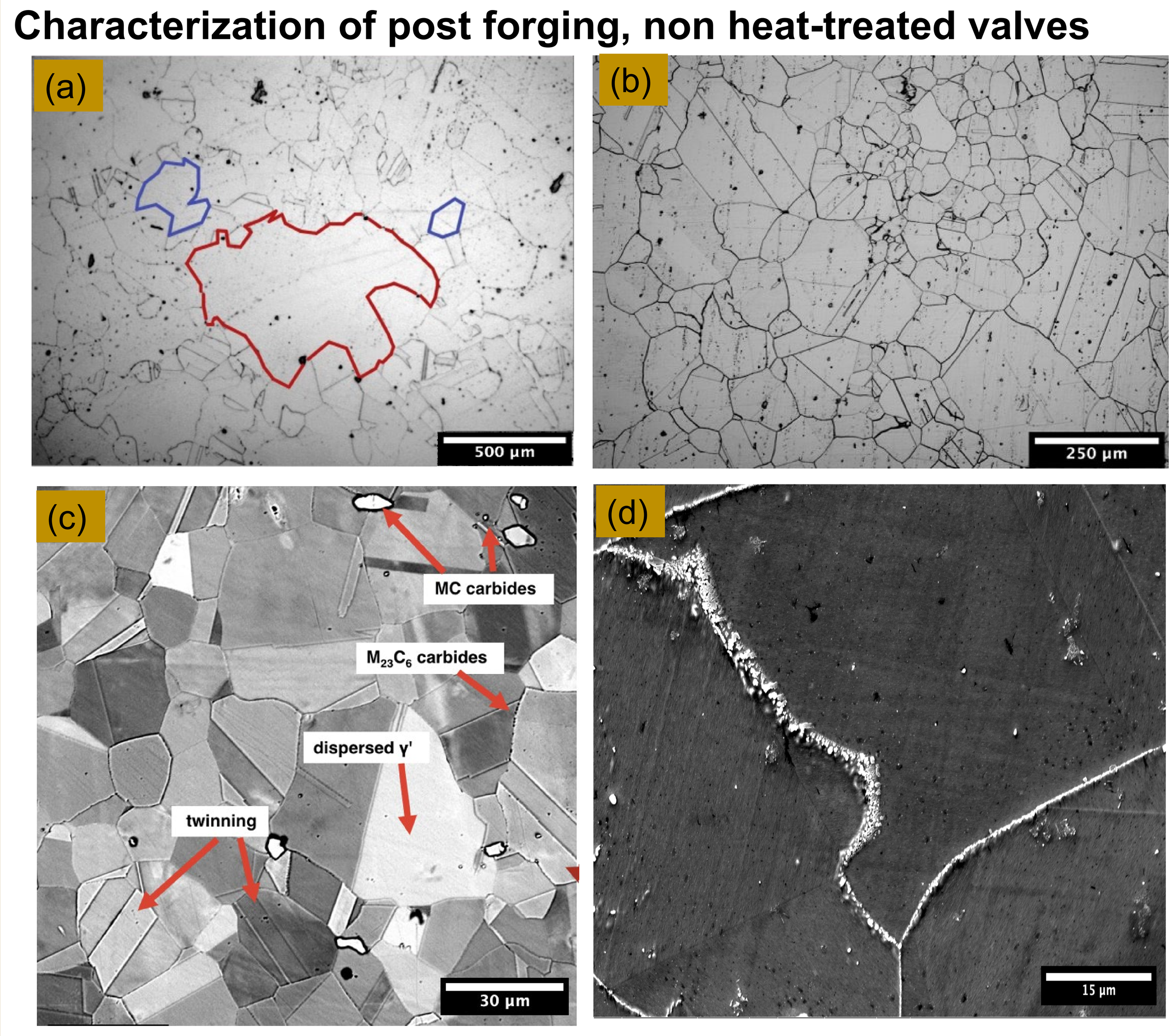
- Bimodal distribution of grains has further decreased near the rounded edge
- Concentrated section of small grains remains near bottom center of sample

Table 2: Grain size characterization in each heat treatment stage at rounded edges.

Case #	Max Grain Diameter (µm)	Distance from Rounded edge (µm)	Min Grain Diameter near max grain (µm)	Grain Size Range (µm)
As-forged				
1	182	368	4	5-200
3	269	301	11	5-300
5	237	281	3	1-300
Solutionizing				
1	406	237	11	10-400
3	293	149	14	10-300
5	251 (longitudinal) 58 (latitudinal)	254	5	5-300
First Aging				
1	326	444	11	10-350
3	402	737	25	5-400
5	336(longitudinal) 65 (latitudinal)	125	12	10-350
Second Aging				
1	399	688	17	10-400
3	386	127	14	10-400
5	379	681	16	10-400

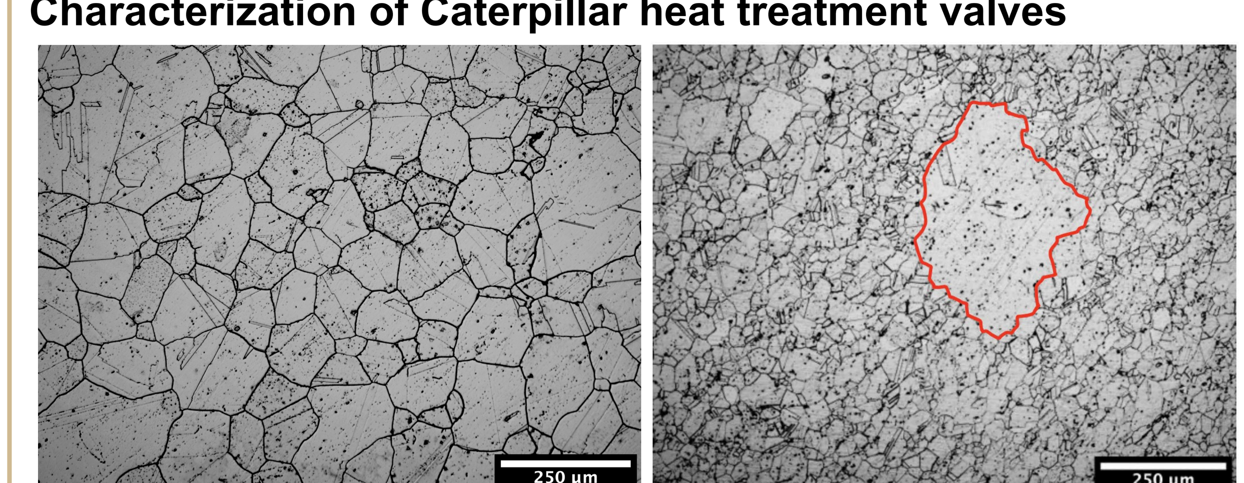
As Received Sample Characterization

Characterization of post forging, non heat-treated valves



- Bimodal grain distribution on valve rounded edges (a)
- Normal grain distribution on valve stem (b)
- Characteristic features of Inconel 751 Microstructure (c)
- Representative carbide morphology along grain boundary (d)

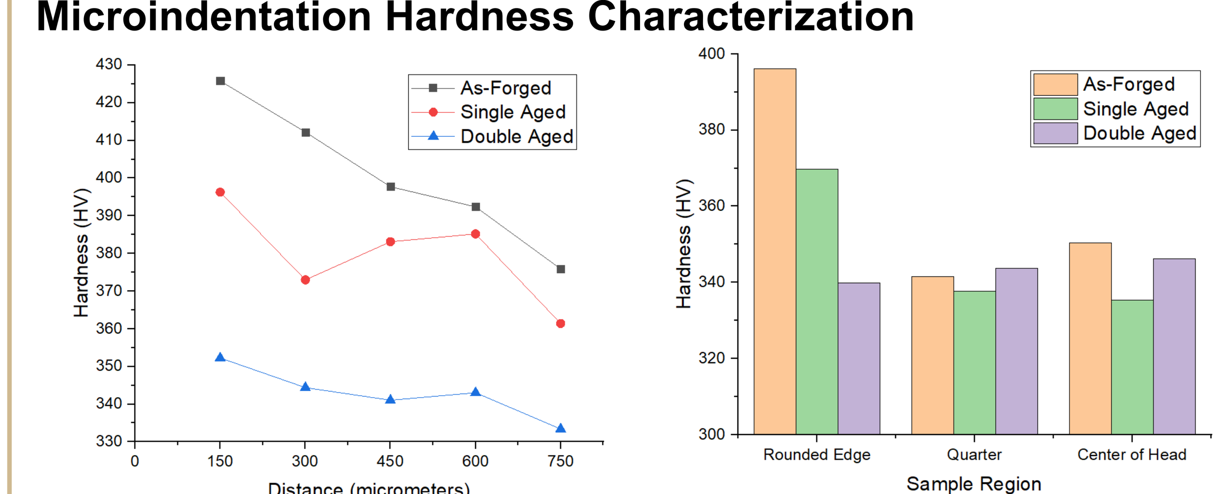
Characterization of Caterpillar heat treatment valves



Case 2, valve Supplier A, Material Supplier A, Forging Process A
- Least bimodal grain distribution

Case 7, Valve Supplier B, Material Supplier B, Forging process B
- Most bimodal grain distribution

Microindentation Hardness Characterization



Hardness values of heat treated samples vs. distance from rounded edge

Comparison of pre, mid, and post treatment hardness values

- Hardness generally decreases with each heat treatment step
- Local and global trend of decreasing hardness the further from rounded edge

Conclusions

- In as-forged samples, along the rounded edge, grains are stretched and elongated in the flow direction. The stretched grains lose their anisotropic shapes and become large grains with non-elongated orientations after heat treatment.
- As the heat treatment progresses, the large grains at the rounded edge shrink and small grains near the center of the valve head grow.
- Peak and overall hardness is highest near the rounded edge. This decreases and becomes more uniform with each heat treatment step
- The experimental heat treatment reduces and normalizes the bimodal grain size distribution but does not fully eliminate it.
- Among the Caterpillar heat-treated valves, case 2 has the least bimodal grain distribution, and case 7 has the most.
- **Recommendations**:
  1) Investigate valve forging process conditions including strain, strain rate, and workpiece temperature during forging on grain size and shape outcomes
  2) Further analyze the effects of intermetallics and carbides on grain growth

Acknowledgements

Caterpillar: Diana Clute, James Peltier, Adam Patrick, Walter Isenberg, Jeff Jansen
Faculty Advisor: Dr. David Johnson
Course Instructors: Dr. Nikhilesh Chawla, Dr. David Bahr
Purdue MSE Staff: Dr. Talukder Alam, Jenni Fifer, Aury Ku Blanco
Special Thanks to Gary George