Project ID: LM045

Materials Characterization Capabilities at the HTML: Surface/Sub-surface dislocation density analysis of forming samples using advanced characterization techniques

2011 DOE Vehicle Technologies Annual Merit Review and Peer Evaluation Meeting

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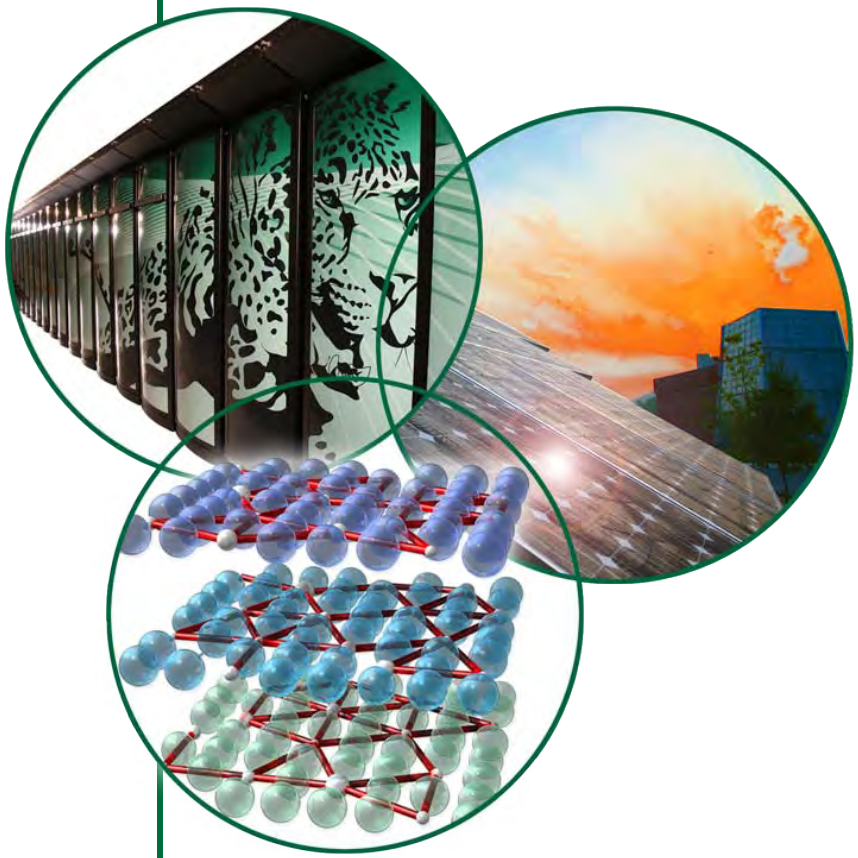

U.S. DEPARTMENT OF ENERGY

OAK RIDGE NATIONAL LABORATORY
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The HTML User Program: Background

- The High Temperature Materials Laboratory is a National User Facility that supports the missions of DOE, EERE and the Vehicle Technologies Program in particular, by working with industry, universities, and other national laboratories to develop energy-efficient technologies that will enable the U.S. to use less petroleum. The HTML is organized into six user centers, which are clusters of highly skilled staff and sophisticated, often one-of-a-kind instruments for materials characterization.
- Access to the HTML User Program is provided through the HTML User Program proposal process. Research proposals are reviewed by a committee and approved based on scientific merit, relevance of the proposed research to the mission of DOE's Vehicle Technologies Program, feasibility, and non-competition with the private sector. Projects have a well-defined scope, and research is completed within 24 months and often involves multiple user visits to the HTML.
- Both nonproprietary and proprietary research is conducted within the HTML User Program. There are generally no charges for nonproprietary research projects, and users conducting nonproprietary research must agree to submit research results for publication in the open, refereed literature. A nonproprietary project is complete when research ends, accompanied by the required publication in the open literature and/or presentation at a professional conference. For proprietary research, the user owns the research data, and all costs at the HTML are paid by the user based on DOE guidelines for ORNL cost recovery.

The HTML User Program – FY2010 Activity

During FY2010, the HTML User Program collaborated with 18 companies, 25 universities, and 6 national laboratories on 68 user projects addressing critical technical barriers to achieving the goals of DOE's Vehicle Technologies Program. There were 96 researchers, 63% of them first-time users, who visited the HTML for a total of 716 research days.

The HTML User Program FY2010 budget was $5,312,400 and allocated as follows:

- Capital equipment: $881,959
- Operations: $4,430,441

Users cost-share their HTML user projects through:

1) direct involvement with HTML staff members during the development of the user project;
2) funding their time and travel to the HTML to perform research;
3) cost of materials provided by the user or the research performed prior to the user project;
4) collaboration with HTML staff members to analyze the data and publish the results.

The HTML also supports the education and preparation of the next generation of scientists and engineers. During FY2010, students and professors from 25 universities participated in the HTML User Program. Five of those students earned their Ph.D. degree and one earned her M.S. degree based in part on research they conducted through the HTML User Program.

Relevance to the VT Program

- The Vehicle Technologies Program funds the operation of the HTML User Program to maintain world-class expertise and instrumentation capabilities for materials characterization to work with industry, universities and national laboratories toward the goals of the Vehicle Technologies Program. The HTML User Program capabilities at the Oak Ridge National Laboratory support the activities of the Vehicle Technologies Program's subprograms in Lightweight Materials, Propulsion Materials, Energy Storage, Solid State Energy Conversion, Combustion & Emissions Controls, Power Electronics & Electric Motors, and Non-Petroleum Fuels.
- During FY2010, the HTML User Program managed **17** characterization projects in Lightweight Materials, and this poster presentation highlights ***one*** of them. The Timken Company user project in this poster presentation is focused on characterizing the crystallographic texture, dislocation motion and density in flow-formed material. This project will increase the understanding of the flow-forming process and aims to improve the efficiency of existing manufacturing processes. The subject of this project addresses technical barriers of cost and manufacturability.

The Timken Company User Project: "Surface/Sub-surface Dislocation Density Analysis of Flow Forming Using Advanced Characterization Techniques"

TIMKEN
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Timeline

- Start date: 01/01/2010
- End date: 01/01/2012
- % complete: 71%

Budget

- Included in the user center allocations from the annual budget of the HTML User Program; users cost-share as noted on slide #3.

Barriers

- Cost ➔ Energy savings for production
- Manufacturability ➔ Improved technology
- Lack of knowledge of advanced materials ➔ properties and performance characteristics

Collaborators

- **Users:**
  Vikram Bedekar and Praveen Pauskar
  The Timken Company
  Rajiv Shivpuri, The Ohio State University
- **HTML Staff:** Jane Howe, Thomas Watkins
  Larry Walker and Dorothy Coffey

Collaborations

As a DOE User Facility, the HTML User Program is collaborative in nature. Potential users are assisted with the proposal submission process as necessary, and all research is hands-on with direct involvement from both user and HTML User Program staff researchers. The DOE-required publication of results for non-proprietary projects is also a collaborative effort.

Collaborators on the user project reported in this presentation:

The Timken Company

Vikram Bedekar, Senior Development Engineer
Praveen Pauskar, Senior Specialist
Rajiv Shivpuri, Professor (The Ohio State University)

High Temperature Materials Laboratory

Jane Howe, Thomas Watkins, Larry Walker, Dorothy and Coffey

The Timken Company User Project: Milestones

An HTML User Program FY2011 milestone is to complete three user projects on the characterization of lightweight and lightweighting materials.

Project ID	Organization	Project	Status
2010-018	Atriax Components Inc.	Characterizing heavy vehicle compressor components: Mg and Al castings and Al-MMC cylinder liner interface	Completed ✔
2010-027	Virginia Commonwealth University	Characterization of lightweight materials for automotive applications	Completed ✔
2010-028	University of Alabama-Birmingham	Effect of chemistry on the transformation characteristics of metastable austenite in intercritically austempered ductile iron for automotive applications	Completed ✔

This presentation reviews one of 17 HTML User Program projects active in FY2010 that focused on the characterization of lightweight materials.

Project ID	Organization	Project	Due Date
2010-009	The Timken Company	Surface/Sub-surface dislocation density analysis of forming samples using advanced characterization techniques	1/12/2012

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Project Objectives & Relevance to Vehicle Technologies Goals

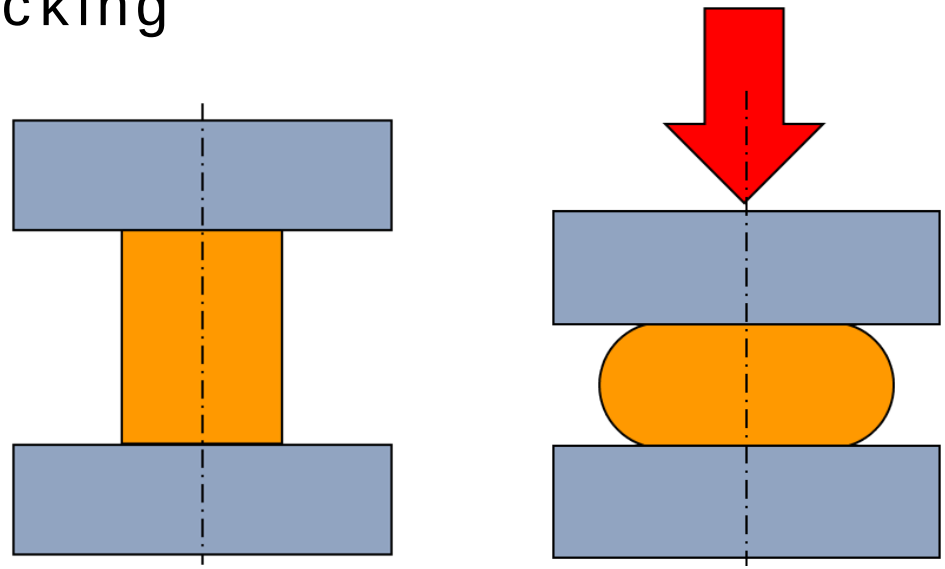
- The Timken Company's user project is focused on developing a greater understanding of why the flow-forming process is able to achieve very large deformations without cracking the material. By understanding the fundamentals of these deformation processes, it will be possible to develop cost-competitive manufacturing processes of automotive assemblies from advanced lightweight materials.
- Prohibitively high cost of finished materials is the greatest single barrier to the market viability of advanced lightweight materials for automotive and commercial vehicle applications. This user project investigates a process that could reduce the cost of manufacturing components for automotive structures using lightweight materials.

The Timken Company User Project: "Surface/Sub-surface Dislocation Density Analysis of Flow Forming Using Advanced Characterization Techniques"

TIMKEN
Where You Turn

Background: Conventional COLD forming of carbon 1050 steel can only achieve ~60% reduction ratio without cracking

Upset forging

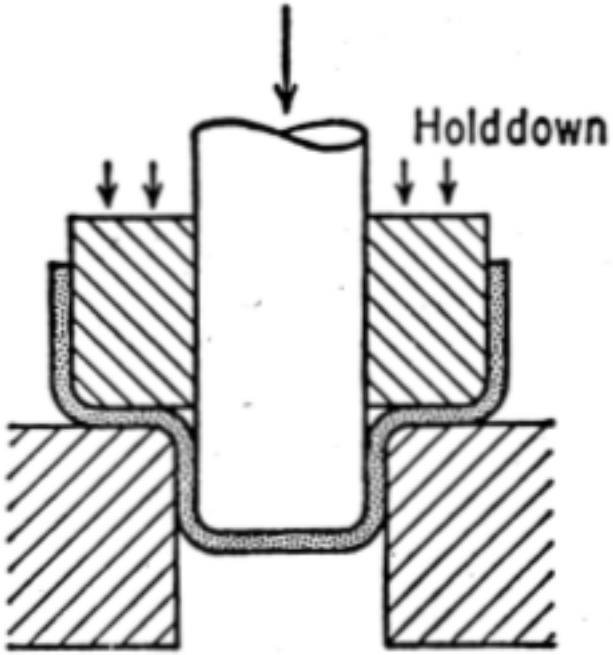


- In standard cold-forming processes, like upsetting, the dislocation density peaks, and at 75% of wall thickness reduction cracking occurs.
- Additional cold working requires a multi-step forming process with intermediate stress relief annealing.

The Timken Company User Project: "Surface/Sub-surface Dislocation Density Analysis of Flow Forming Using Advanced Characterization Techniques"

TIMKEN Where You Turn

Background: FLOW forming process can achieve 90% wall thickness reduction without evidence of cracking



Dieter, *Mechanical Metallurgy*, 3rd edition, 1986, pp. 670,674

- Greater reduction ratios improve manufacturing efficiencies.
- Example parts at Timken show up to 90% reduction ratio without cracking.
- Recovery and recrystallization occur naturally.
- Hypothesis: forming proceeds as dislocation density increases.
- Crystallographic analysis is needed to understand the dislocation travel and density in flow-formed material.

The Timken Company User Project: "Surface/Sub-surface Dislocation Density Analysis of Flow Forming Using Advanced Characterization Techniques"

TIMKEN Where You Turn

Approach: Use advanced characterization tools

- Identify un-cracked formed samples of alloys at Timken facilities
- Microstructural characterization of un-cracked formed alloys using a variety of tools (TEM, FIB, XRD, and SEM)
  - Focused ion beam (FIB) technique: to prepare cross-sectional formed surface/ subsurface samples.
  - Scanning transmission electron microscopy (STEM): microstructure, crystallinity, dislocation densities of the subsurfaces.
  - Scanning electron microscopy (SEM): microstructure and microscopic crystallographic texture.
  - X-ray diffraction (XRD): macroscopic crystallographic texture after forming.
- The unique combination of advanced equipment and expertise in one location positions the HTML User Program to help Timken improve existing product/process technology, which will ultimately result in improved component life and energy savings.

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Approach: Current samples formed at 60-90% reduction ratios

Conditions

- Alloy chemistries (wt%)
  - Carbon steel 1050: 0.50 C/ 0.7 Mn/ bal. Fe
  - Stainless steel 17-4PH: 17 Cr/ 4 Cu/ 4 Ni/ bal. Fe
  - Inconel 718: 20 Cr/ 3 Mo/ 5 Nb/ 50 Ni/ bal. Fe
- % reduction ratio (0, 60%, and 90%)

The Timken Company User Project: "Surface/Sub-surface Dislocation Density Analysis of Flow Forming Using Advanced Characterization Techniques"

TIMKEN
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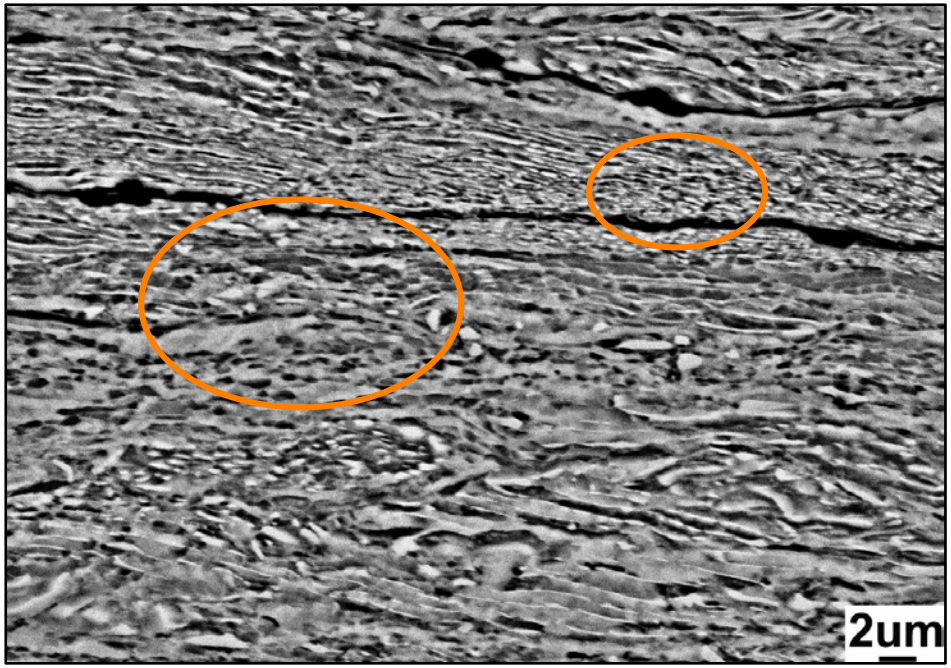
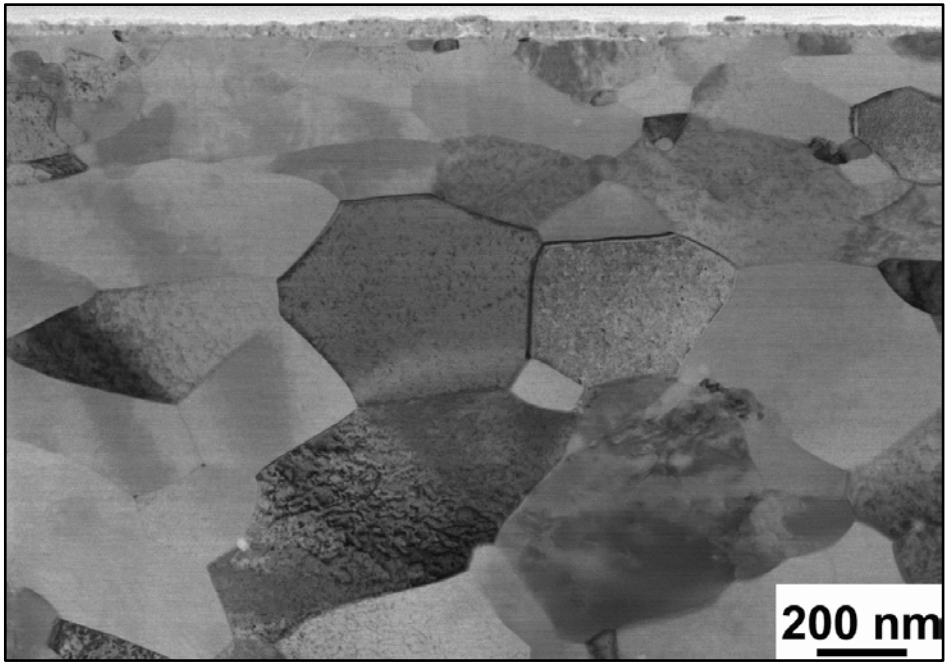
Accomplishments: A detailed study of cold work subsurfaces using TEM, SEM and XRD

carbon steel 1050 after 66% reduction



Carbide size – 200nm to 1.4μm

Microstructural transformation due to severe plastic deformation



Equiaxed grain size~300nm, 1:1 aspect ratio

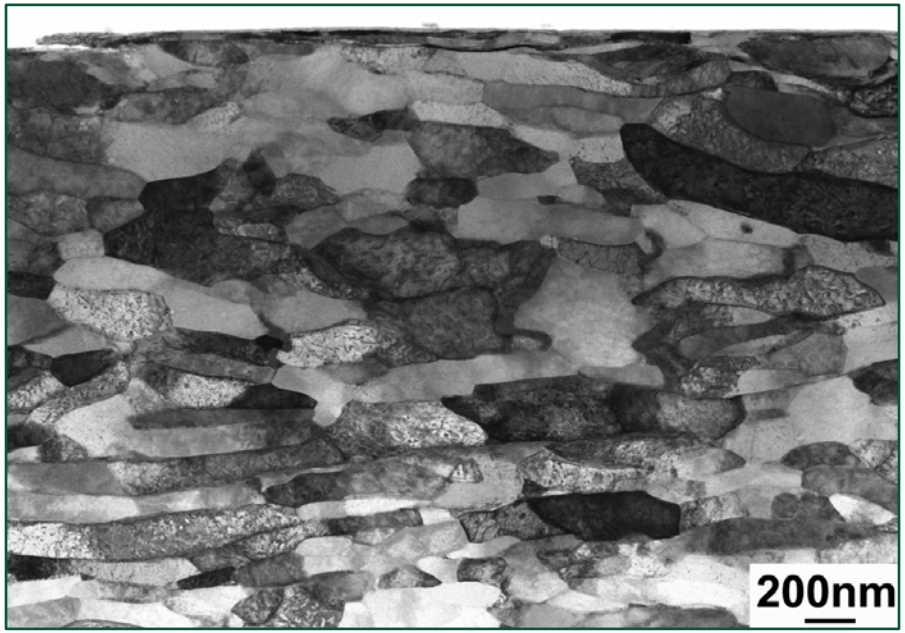
Equiaxed grain structure due to dynamic recrystallization

The Timken Company User Project: "Surface/Sub-surface Dislocation Density Analysis of Flow Forming Using Advanced Characterization Techniques"

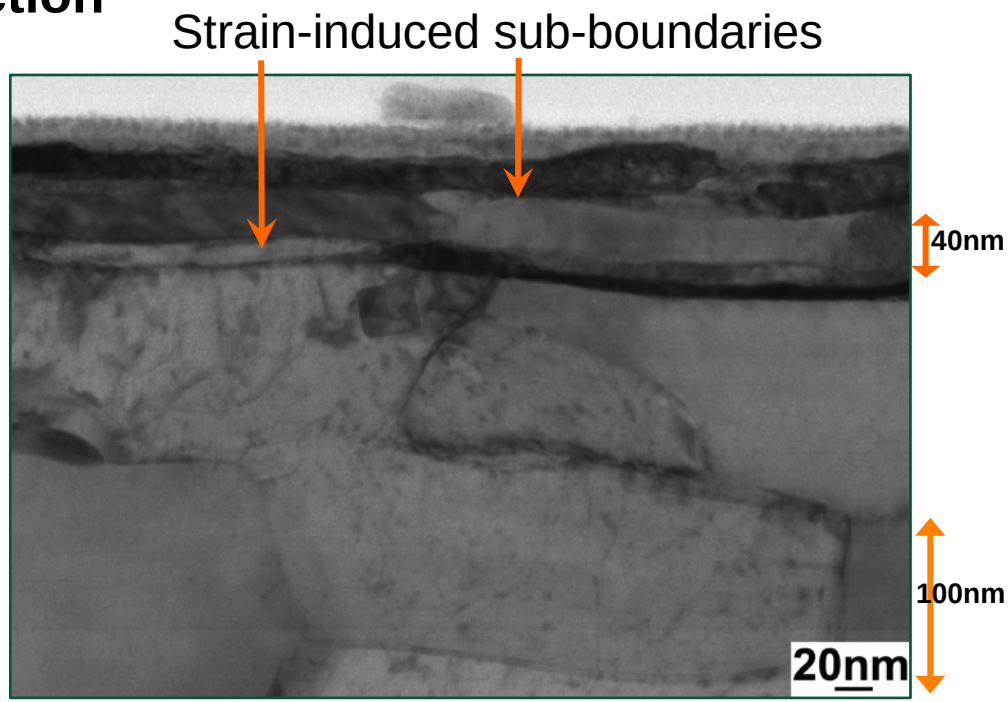
TIMKEN
Where You Turn

Accomplishments: A detailed study of cold work subsurfaces using TEM, SEM and XRD

carbon steel 1050 after 90% reduction



Ribbon-like substructure
6:1 aspect ratio

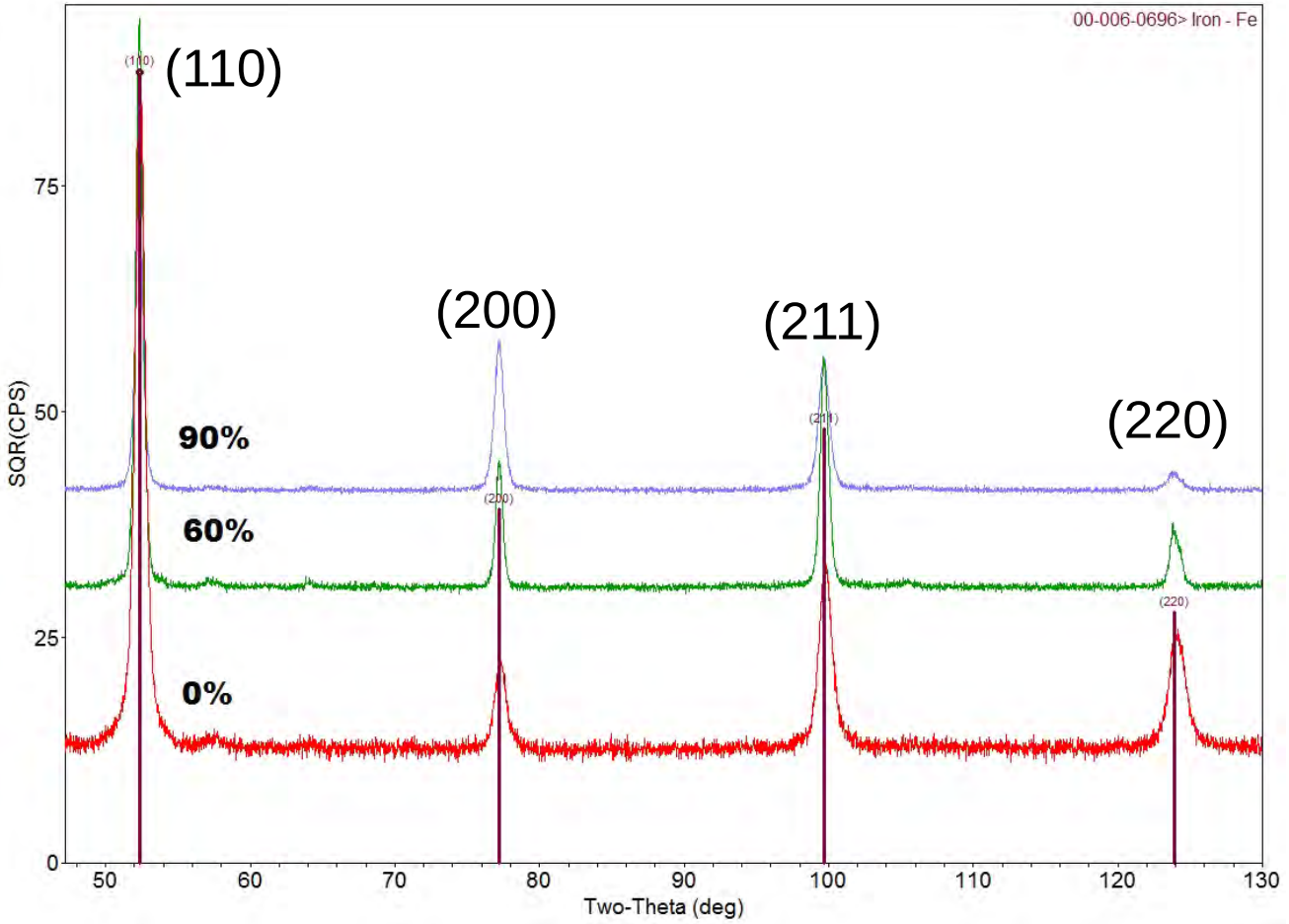


Development of ultra fine (sub) grain cell structure at 90% deformation

The Timken Company User Project Accomplishments

XRD relative intensity changes indicate a change from random to preferred orientation caused by deformation.

carbon steel 1050



The Timken Company User Project Accomplishments

XRD shows the intensities of the (200) and (211) peaks in carbon steel 1050 increase with deformation relative to the (110) and (220) peaks.

	Relative Peak Counts		
hkl	0%	66%	90%
110	100	100	100
200	5	14	46
211	13	32	41
220	7	7	5

- **(200) is an immobile slip plane that causes strengthening and crack initiation sites, due to the developed preferred orientation/texture.**
- **The possible reaction could be: a/2 [111] + a/2 [-1-11] → a[200] ****

** Dieter, *Mechanical Metallurgy*, 3rd edition, 1986, pp. 159-60

The Timken Company User Project Accomplishments

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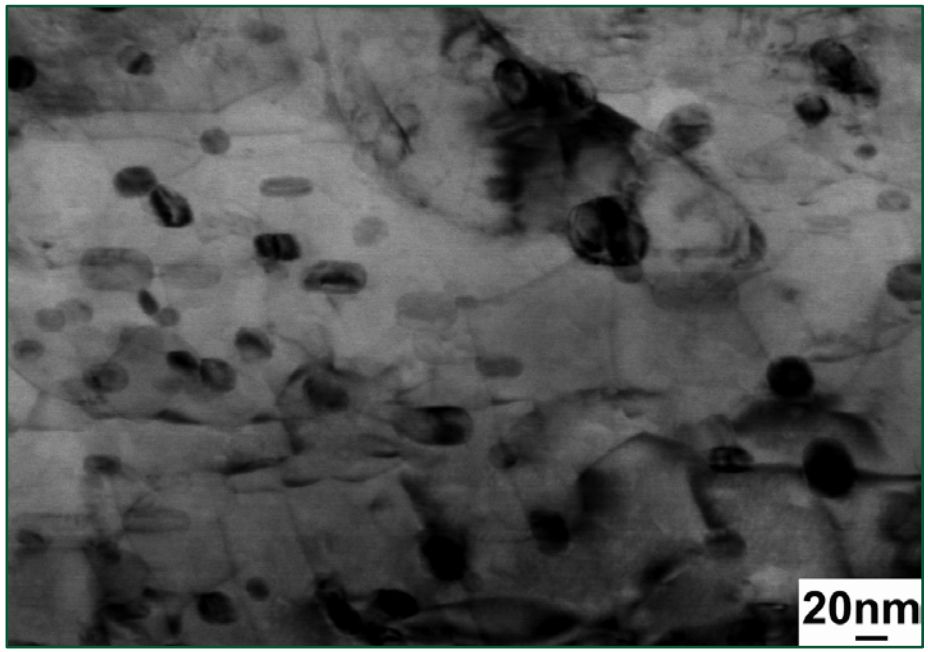
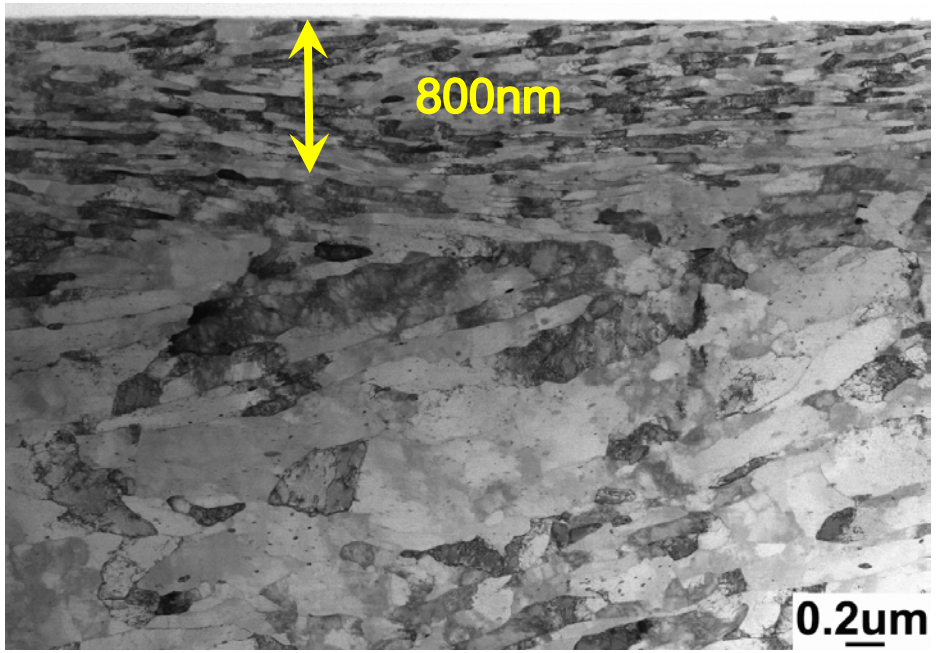
A detailed cross-sectional TEM study of cold-work sub-surfaces shows dislocation pinning and grain refinement.

stainless steel 17-4PH after 60% reduction



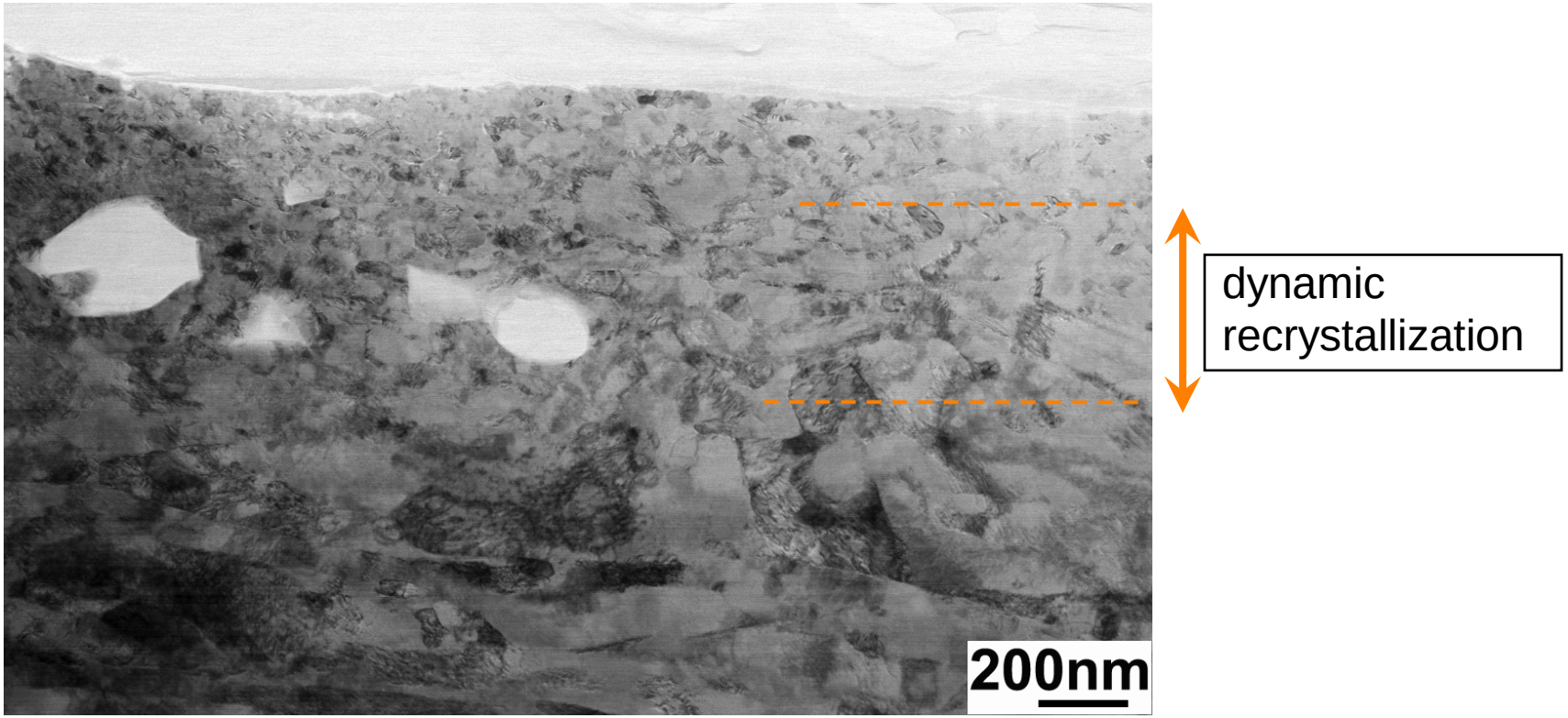


Bulk (> 2um depth) Dislocation pinning at 17-4PH due to Cu precipitates

Subsurface (<800nm): Grain refinement due to severe plastic deformation

The Timken Company User Project Accomplishments

Inconel 718 specimens showed near-surface dynamic recrystallization.



Hardness increased from 10HRc-pre-formed to 50HRc due to severe strain hardening and strain-induced martensitic transformation.

The Timken Company User Project: Summary

- The Timken Company participated in the HTML User Program to study flow-formed carbon steel 1050, stainless steel 17-4PH, and Inconel 718 at deformation ratios of 60-90% using a combination of characterization techniques (TEM/FIB, XRD, SEM).
- The research helped clarify the prevalent microstructural features, strengthening mechanism, preferred orientation, dynamic recrystallization, evolution of grain size, and dislocation pinning mechanism due to dispersion strengthening. For example, in stainless steel 17-4 PH, the hard copper precipitates pin dislocations, causing severe strain hardening.
- The Timken Company was able to take advantage of a wide array of materials characterization capabilities available to industry users through the HTML User Program, which maintains world-class expertise and unique instrumentation gathered in one convenient location. The results from this investigation lay the groundwork for cost-effective manufacturing process with the potential to enhance post-forming mechanical properties and enable the increased use of lightweight materials in automotive structures.

The Timken Company User Project: Future Work

- HTML User Program projects are expected to be completed within 24 months, and The Timken Company's project is on schedule.
- The experimental phase of this project has been completed, and its research objectives have been met. Data analysis is in progress and will be followed by the preparation and submission for publication of a manuscript.
- As it has done several times in the past, The Timken Company plans to continue its collaboration with the HTML through new projects submitted to the HTML User Program that help achieve goals set by the DOE Office of Vehicle Technologies.