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Author(s): Olaf Engler (MST-CMS)
John Bingert (MST 6)
Hans-Rudolph Wenk (UCB)
Mike Stevens (MST 8)

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Texture Science and Technology

Olaf Engler, John F. Bingert, Rudy Wenk (Dept. Geology and Geophysics, UC Berkeley), Mike F. Stevens *

Abstract

This project is directed at advancing research and development in texture and anisotropy at Los Alamos. We are recognized as a national and international leader in texture and anisotropy research. This recognition is based on our understanding involving both quantitative texture analysis and the understanding and modeling of processes under which texture develops. In addition to these resources, we have available the full troika of texture measurement techniques, namely, x-ray, electron diffraction, and neutron diffraction. The goals of this project were (1) to increase the utilization of texture and anisotropy both within and without the Laboratory programmatic, basic, and industrial related efforts; (2) to seek to improve our texture measurement and modeling capabilities; and (3) to maintain our recognition as an international leader through basic research.

These goals were accomplished through the formation of a coherent focus on texture directed through the CMS to coordinate texture efforts at the Laboratory as well as advancing the field in analysis, measurement and interpretation. The "texture focus" has essentially four functions: One, to manage the physical measurement systems; two, to coordinate human resources at the lab; three, to serve as a resource for both external and internal users; and four, to advance the field of texture analysis at all levels and keep it at the forefront.

Background and Research Objectives

Similar to the false association of materials science with the mere production of micrographs, texture analysis is often perceived as the production of pole figures. However, texture analysis is much more than pole figures and orientation distributions. Texture and anisotropy determines a material's properties and have become a specification requirement in metallurgy. A prime example is in the production of suitable aluminum sheet for beverage cans, which has been one of the main motivations for texture research in the United States. Texture is also being used as a process control specification in the production of tantalum plate for ballistic applications. Texture is crucial in the characterization of thin films in electronic devices. It determines the performance of superconductors. The strength and failure of structural ceramics and polymers depends largely on the crystallite orientation. In earth sciences, seismic anisotropy has become one of the important topics of research. It controls movements within the earth and is essential for interpreting seismic reflection data.

Texture analysis is an interdisciplinary field and not just with respect to the materials. The

quantitative study requires interaction between materials scientists, physicists, mathematicians, statisticians, crystallographers, mechanics and others; measurements rely on optical microscopy and, particularly, X-ray, neutron and electron diffraction. This diversity puts texture analysis beyond a single person operation. It has to rely on a variety of experts, a wide range of users and access to modern facilities.

Faster computers, new graphics environments, new measurement hardware and other developments have enabled textures to be more readily measured with more detail and precision than ever before. An excellent example is in measuring microtextures with the scanning electron microscope. This new instrumental capability gives the materials scientist a wealth of data that was previously unavailable. However, collection of data is one matter, without quantitative processing and imaginative interpretation it does not serve much purpose. Texture analysis is very complex, each case has to be analyzed individually and a black box approach, e.g. operated by a technician, commonly fails. This is one of the reasons why many laboratories leave the field after initial enthusiasm. With new measurement systems the data is even more complex and methodologies for interrogating that data to gain understanding or to solve technical problems are still in their infancy. Nonetheless, texture analysis carries enormous technical and scientific benefits with application to a wide variety of problems.

Texture research in the United States is generally quite fractured and therefore lagging behind Europe and Asia in spite of early US pioneers such as Barrett, Hu, Morris, and Roe. In the US, texture research is conducted at a few universities and a handful of industrial and government laboratories to solve specific technical and scientific problems. With the diminishing capability of industrial research labs there is no collective synergy anywhere in the United States. Presently, Los Alamos probably has the highest concentration of scientists with an active interest in texture and anisotropy. Indeed, Los Alamos is recognized as a national and international leader in texture research. This is based on some outstanding research, involving both quantitative texture analysis and understanding of processes under which texture develops. The Center for Materials Science (CMS) has, under the direction of Dr. Kocks and collaborators, developed a system of computer programs, *popLA*, which has become a standard and is used world wide. In addition to human know-how, there are necessary facilities in X-ray, electron and neutron diffraction. Many programs have been sold to Laboratory customers based on texture expertise. Yet, even at Los Alamos, efforts had been accidental and uncoordinated. Thus, there was a distinct window of opportunity to firmly establish Los Alamos as a leader in texture analysis.

Importance to LANL's Science and Technology Base and National R&D Needs

The processing and use of materials with anisotropic properties is becoming increasingly important. Every step in metalworking: from casting molten metal into an ingot and then forming the ingot into a bar or a sheet and then annealing a finished form, changes the texture and therefore the

properties of a material. Strengthening our understanding in texture can have an effect at many different levels. For example, the materials scientist can use texture analysis to gain increased fundamental understanding into deformation; the design engineer can use texture to more accurately predict the effect of the plastic and/or elastic anisotropic response of a textured material on a design component; and the production engineer can use texture to qualify a material for a particular application.

Texture has played and continues to play an integral role in many activities at LANL. Examples include a CRADA with Exxon, where the role of texture and microtexture have been observed to have an impact on stress-corrosion cracking in oil transmission pipe as well as a CRADA with the "big three" automakers and two of the major aluminum producers to come up with low cost aluminum sheet which satisfies material property demands for automobile sheets. In the framework of the joint DOE/DoD munitions technology development program texture has been shown to have a pronounced effect on the performance of ballistic applications.

As previously mentioned every step in metalworking changes the texture and therefore the properties of a material. Thus, any Lab program where material performance plays a significant role can derive benefits from texture analysis: such as improved materials models, more complete materials characterization and improved material processing. Although one mission of the focus team was to advance the field of texture analysis, the primary focus was to advance the use of texture analysis to gain fundamental understanding into material forming and to solve a myriad of engineering and science problems. Texture has been a strong part of materials modeling at Los Alamos. Therefore, it was expected to maintain close contact with the Materials Modeling Project and to enhance the capabilities of materials modeling at Los Alamos.

Scientific Approach and Accomplishments

The main objective of the LDRD project "Texture Science and Technology" is directed at advancing research and development in texture and anisotropy at LANL. To conduct texture measurements on different scales, LANL has available the full troika of texture measurement activities, namely, electron, X-ray and neutron diffraction. One of the main goals of the current project was to improve our texture measurement capabilities (Sec. 1). For that purpose, a project to build the high count-rate general purpose time-of-flight neutron diffractometer 'HIPPO' (for High Pressure – Preferred Orientation) has been advanced at LANSCE. The HIPPO diffraction spectra contain information on microstructure, internal stresses and preferred orientation of crystals in aggregates and new methods have been developed to extract information efficiently and quantitatively. Furthermore, the EBSD-microtexture capability were brought on-line and have been intensely used for a variety of projects since.

The recognition of LANL as a national and international leader in texture and anisotropy research is based on our understanding of mechanisms under which textures develop. In order to

7expand our knowledge in texture evolution during materials processing, numerous experiments have been conducted whose main achievements are summarized in Sec. 2.

LANL has developed a set of computer programs for modeling the evolution of texture during deformation as well as the anisotropic mechanical response for textured materials. To improve our modeling capacities, several projects to model recrystallization have been conducted (Sec. 3).

1. Texture Measurement Capabilities

1.1 HIPPO

Electrons and x-rays are used routinely in every materials science laboratory to investigate properties of materials. Even though neutrons have distinct advantages they are rarely applied. The main reason is that access to neutron sources, such as nuclear reactors and linear accelerators has been very limited. The University of California has advanced a project to build a high count-rate general purpose neutron diffractometer at LANSCE, which has become known as ‘HIPPO’ (for HIgh Pressure – Preferred Orientation) and was selected for rapid construction under the DOE-sponsored SPSS Project and is presently being built at Los Alamos and UC Berkeley. The HIPPO time-of-flight neutron diffractometer is designed to study properties of polycrystalline materials (including powders) and liquids. Of particular interest is the investigation of small (1mm^3) and large (2cm^3) sample volumes at high (2000K) and low (10K) temperatures at high pressure (20GPa), and in different atmospheres. A flexible sample environment (large 75 cm diameter sample well) will accommodate ancillary equipment such as goniometers, furnaces, cryostats, straining stages, high-pressure cells, etc. The diffractometer will have main applications in the fields of phase transformations, high pressure research, polycrystal anisotropy (texture-strain-stress) and complex materials (crystallography). It will be possible to study the dynamics of reactions, recrystallization and deformation of bulk anisotropic samples at a wide range of temperature and pressure conditions. No existing instrument, world-wide, has this capability.

Advantages of neutron diffraction are mainly low absorption (applicability to large coarse samples, environmental stages) and high spectral resolution [1]. In cooperation with scientists at LANSCE and IPNS new methods of texture analysis have been developed which take advantage of the unique properties of TOF neutrons [2,3,4]. First 2D position sensitive detectors were explored but ultimately individual detector banks as implemented on powder diffractometers were preferred. This research and parallel investigations by Von Dreele [5] provided the basis for the design of the HIPPO diffractometer.

A diffraction spectrum can be explained on the basis of the incident beam, the physics of the diffraction process, instrument parameters (such as resolution), background and the characteristics of the material. These characteristics include crystal structure (lattice, atomic positions and thermal vibrations), the orientation distribution of crystallites in a polycrystal and microstructure (grain size

and shape) and elastic strain. The crystallographic Rietveld method is an elegant approach to extract this broad information and was particularly expanded to include texture analysis [6] and a multi-platform software, written in Java, is presently in a general state of testing. The goal has been to produce a user friendly quantitative software system that is accessible to the average materials scientist who does not have much knowledge about crystallography and texture analysis.

The method has been applied to some systems which were chosen because of their general interest and because of specific properties that make them useful test cases. The first case is a two-phase metal composite Cu-Fe to evaluate changes in texture, microstructure and strain during deformation and recrystallization. The second case is bulk high temperature superconductors with extremely sharp textures and very complex diffraction spectra. The third case is peridotite, a polymineralic rock with weak to moderate textures. The components olivine (orthorhombic) and pyroxene (monoclinic) have low crystal symmetry. Peridotite is important to geophysicists because its deformation causes seismic anisotropy in the Earth.

1.2 EBSD

Nowadays there is a selection of techniques available to analyse the texture, i.e. the crystallographic orientation, of materials. The well-established techniques of X-ray or neutron diffraction, known as *macrotecture*, are now supplemented by methods whereby individual orientations are measured in both transmission and scanning electron microscopes and directly related to the microstructure, which has given rise to the term *microtexture*. Microtexture practice has grown principally through the application of electron back scatter diffraction (EBSD) and it is now possible to measure orientations automatically from pre-determined co-ordinates in the microstructure, which is known generically as *orientation mapping*. For that purpose, the sampling area is rastered by controlling the electron beam or the sample stage in the SEM (Philips XL30 FEG) in steps of say 0.1–10 μ m, and for each point the crystallographic orientation is determined by EBSD. This technique – which is commonly referred to as orientation imaging microscopy (OIM[™], registered trademark of TexSEM Laboratories, Inc., Draper, UT) – allows the reproduction of the microstructure of a sample from the crystallographic orientations of the microstructural elements (i.e. grains, subgrains) and yields valuable information on the mutual misorientation between adjacent grains [7]. Within the framework of the present project an OIM capability was established in the Electron Microscopy Facility in order to interrogate the microtexture of materials and correlate spatially resolved texture with properties.

1.3 X-ray texture analysis

Despite the beforehand-mentioned obvious advantages of neutrons (low absorption/high penetration depth) and electrons (spatial resolution) for texture analysis, X-ray diffraction is still the work horse of quantitative texture analysis. In framework of the present project, a new texture measurement system (Scintag PTS) with 4-circle goniometer, X-Y sample translation stage, Peltier-cooled solid-state detector and residual-stress measurement software was installed and brought into full operation for rapid texture measurements.

Furthermore, the existing capabilities to measure X-ray textures were upgraded with new software control which speeds up pole figure measurements by enabling a more efficient scanning procedure [8]. In the standard *equal angular* grid the density of measuring points per area is very inhomogeneous, as the pole figure centre is covered with a much higher density than the outer circles. To cover the pole figure more uniformly, an *equal area* scan can be applied, which reduces the number of measurement points by about one half. Thus, for a given angular resolution, the recording time can be halved or, allowing the same total measuring time, the angular resolution can be doubled.

2. Experimental Investigations

2.1 Correlation between pitting corrosion, orientation, and grain boundary character

Microtexture measurements were also applied to research the correlation between pitting corrosion, orientation, and grain boundary character for variously processed Be [9], Stainless Steel, and Ni samples, and to furnish experimental validation for plasticity modeling of Ta [10,11] and hcp metals.

2.2 Bi-based superconductors

Applications included the investigation of intercolony and intracolony misorientations in Bi-2223 superconductor tapes, in which a preference for low-angle intracolony misorientation distributions was discovered. Conventional x-ray texture measurement and analysis was applied to deformation- and reaction-induced texture development in Bi-based superconductors at both bulk and local scales. Results show sharpening of c-axis alignment upon conversion from 2212 to 2223 stoichiometries, and a mesotexture present in Bi-2223 tape samples. A method to use standard x-ray diffractometers to determine fiber textures was established using HTSC tapes in collaboration with NIST and American Superconductor [12].

2.3 Nucleation of recrystallization at the grain boundaries

After rolling and subsequent recrystallization annealing the textures of most commercial Al-

alloys are composed of two orientations, the cube-orientation $\{001\}\langle 100\rangle$ and the R-orientation $\{124\}\langle 211\rangle$ close to the former rolling texture orientations. Whereas the occurrence of the cube-orientation has been subject to many investigations and is quite well understood, there is a surprising lack in information on the R-orientation although in many textures the R-orientation even has a much higher volume than the cube-orientation. In the framework of the present project the factors affecting the R-orientation – and in particular its strong materials-dependence – have been reviewed [13], resulting in a much better understanding of the formation of this important recrystallization texture component, which is necessary with regard to a comprehensive understanding of the recrystallization during thermomechanical treatment of Al-alloys.

2.4 Influence of dispersoids on the recrystallization of Al-alloys

It is generally known that the recovery and the recrystallization behavior of Al-alloys is strongly affected by the precipitation state (e.g. [14]). Small dispersoids are known to strongly retain both recovery and recrystallization and, thus, retard the progress of recrystallization. Large particles ($>1\mu\text{m}$), on the other hand, generally promote recrystallization by particle stimulated nucleation (PSN; e.g. [15]). In the case of thermomechanically processed Al-alloys, both types of particles may occur (bi-modal particle distribution), resulting in a complex interaction between the large particles and the small dispersoids. In addition, in the case of a supersaturation of solute atoms, new dispersoids may precipitate during the early stages of the recrystallization anneal which further affects the progress of recrystallization. With regard to the influence of different states of precipitation/supersaturation during thermomechanical processing of Al-alloys, the influence of dispersoids on the recrystallization behavior has been analyzed in a ternary Al-Fe-Si model alloy. Different states of precipitation and supersaturation were prepared according to different pre-annealing treatments. The evolution of the microstructure and, particularly, of the crystallographic texture was followed during cold rolling and recrystallization. In dispersion-free sample states, PSN occurring within the deformation zones around the large constituent particles gave rise to fine grained microstructures and very weak recrystallization textures. In the presence of small finely dispersed precipitates, either already present in the as-deformed state or precipitating during the recrystallization anneal, however, a more pronounced cube recrystallization texture and larger grain sizes were obtained. This indicates that the dispersoids selectively suppressed PSN but did not significantly affect nucleation at cube-bands [16].

These results are of great interest e.g. with view to developing competitive Al-alloys for car-body applications. Preliminary results of a study performed in cooperation with Dr. C.T. Necker and Dr. D.A. Korzekwa (both LANL-MST6) on texture evolution, mechanical properties and plastic formability of sheets of both non-age hardening 5xxx (Al-Mg) and age-hardening 6xxx (Al-Mg-Si) alloys after different thermomechanical processings that were provided by Reynolds Metals strongly suggest that the recrystallization textures are indeed governed by the precipitation state. In turn,

appropriate texture control can eventually improve the plastic anisotropy of the sheets.

Annealing of cold rolled, supersaturated Al-1.3%Mn leads to heavy precipitation of fine particles on the as-deformed microstructure. In dependence on the crystallographic orientation of the deformed matrix grains particles with different morphologies – spherical, rhomboidal and plate-like – have been observed. This variation in morphology could be traced back to differences in local misorientation caused by different dislocation substructures in different matrix orientations. Microstructural investigations and in particular SAD-analysis in a TEM were employed in order to characterize the various particles and to determine the possible orientation relationships between the plate-like precipitates and the Al-matrix which may occur during continuous recrystallization of Al-Mn alloys. The variety in orientation relationships is explained in respect to the influence of local misorientations in different matrix orientations caused during deformation and the existence of several low misfits of plane spacings in both phases [17].

2.5 Evolution of through-thickness texture gradients in rolled and recrystallized sheets

Approximation of practical rolling operations by a plane strain state is strongly simplified, since factors like geometrical changes during a rolling pass, friction between roll and sheet surface and strain and temperature gradients upon hot rolling can cause severe deviations from the plane strain condition. Furthermore, these parameters depend on the distance from the surface of the rolled sheet, giving rise to different strain states, and hence to different rolling textures, at different through-thickness layers of the sheet, which in turn may strongly affect the plastic properties of the rolled products.

In cooperation with Prof. M.Y. Huh and coworkers (Korea University, Seoul), causes and formation mechanisms of through-thickness texture gradients have been analyzed in rolled sheets of copper and low carbon steels both in the as-deformed state and after recrystallization [18,19]; other materials (Al, austenitic steels) are currently still under investigation. In order to obtain different local strain states and, hence, different deformed microstructures, specimens were deformed by cold rolling with and without lubrication. The effect of these different rolling procedures – and hence of different local strain states through the sheet thickness – on the evolution of microstructure and texture during rolling and recrystallization was studied by X-ray texture analysis and by microstructural and -textural observations in TEM and SEM, including EBSD-measurements. A simple model has been developed to understand the typical shear texture observed at the surface of the inhomogeneously rolled sheets. This should provide us with new insight into the formation of through-thickness texture gradients and their impact on the mechanical properties on sheet products.

3. Modeling Capabilities

Based on the assumption that recrystallization textures evolve by a growth selection of distinct

grains out of a limited spectrum of preferentially formed nucleus orientations, the recrystallization textures can be simulated by the multiplication of the probability of nucleation of the orientations with the probability of their subsequent growth [20]. This model in combination with an approach to derive the number of nuclei forming at the various activated nucleation sites [21] now permits simulation of the recrystallization textures of Al-alloys in dependence on both microstructural characteristics and processing parameters [22].

As an example to demonstrate the predictive power of this model, the recrystallization textures of various Al-alloy deformed at a variety of strains, strain rates and deformation temperatures – i.e. at different values of the Zener-Holloman parameter Z – were simulated [23]. The model provided a very satisfactory simulation of the corresponding recrystallization textures by reproducing the characteristic shift from a cube-recrystallization texture towards a weak texture as obtained in the case of PSN with increasing Z . Thus, the model is able to simulate the recrystallization textures of Al-alloys for a wide range of microstructural characteristics and processing parameters during the thermomechanical processing of Al-alloys, which can be used to predict and eventually to improve the properties of the final Al-sheet products.

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