From: LABSYS/MICHAEL
Subject: Incorrect ODF analyis
To: PAUL
X-To: Paul
Date: 19 Apr 99 10:24:52
X-pmrqc: 1

OK Incident Report?

Paul

I read the reports in question have checked the inverse pole figure, the best I can for several projects.

605-7279 Is O.K. This is the project which Perry and Keith billed hours to.

74-8057 Is definitely wrong. Based upon the Inverse pole figures and the (0002) pole figure an extra 90 degree rotation was performed

674-8321
-8235
-8151
-8015 all had spotty pole figures with a "fibre" texture making it difficult to judge by the pole figure if a mistake was made, but the inverse pole figures are not the same in the transverse and reference direction. Since Perry did the work on these I would assume that these were rotated as well, making the inverse pole figures denoted by "1" and "2" incorrect and features in "3" skewed.

Lee Sanitation

388-7712 Is difenitely wrong

Since WPAFB will be informed by Allied signal I will verify that the incorrect rotation was performed. If so I will begin to regenerate the data at least in the reports above.

Also, how are we to inform the customers of the mistake made.

Also, since I need to post my hours daily, how do I post the one I am spending lookin at these projects?

Mike

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A/2

LAMBDA RESEARCH
QA INCIDENT REPORT

Procedure: G E Nuclear Sample Prep and Texture Analysis of Zircalloy-2 Plate and Tube Samples	Procedure No.: 3P 1066 (3P1016)
Equipment: 11	Reported by: Mark Kelly
Project No.: 5-8728, 5-8796	Responsible Tech: CB, MK
File No.: 986985-01	Date: 8/25/99
Specimen ID: 986985-01	Time: 4:30

Description of problem: See attached.

Describe cause: See attached.

Describe solution: See Attached

Actions to be taken: See Attached

Supervisor Signature: Mark J. Kelly Date: 8/26/99

Employee Signature: Chris Berger Date: 9/1/99

Director Signature: Date:

Approved By:	[illegible] 12/23/94 (Author)	[illegible] 12/23/94 (QA Administrator)	[illegible] (Director of Research)	5F2000.02 Revised 12.01.94

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From PM 8/17/99

LAMBDA RESEARCH
QA INCIDENT REPORT

Procedure: GE Nuclear Sample Prep and Texture Analysis of Zircaloy-2 Plate and Tube Samples	Procedure No.: 3P1066 (3P1016)
Equipment: H	Reported by: Mark Kelly
Project No.: 5-8728, 5-8796	Responsible Tech: CB, MK
File No.: 986985-01	Date: 7/15/99
Specimen ID: 986985-01	Time:

Description of problem: See Attached.

Describe cause: See Attached.

Describe solution: See Attached.

Actions to be taken: See Attached.

Paul:

1) Is this OK?
2) Should I or someone else review the older pole figure data to see if its OK?
3) Let me know if there should be changes. (Sorry to bother you with this today.) Mark.

Supervisor Signature:	Mark Kelly	Date:	7/16/99
Employee Signature:	Chris Berger	Date:	7/16/99
Director Signature:		Date:	

Approved By:	PMHeson 12/23/94 (Author)	PMHeson 12/23/94 (QA Administrator)	[signature] (Director of Research)	6F2000.02 Revised 12.01.94

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Lambda Research QA Incident Report, 7/15/99, Attachment 1.

Description of Problem: A specimen from sample 986985-01 was not prepared properly. This introduced errors in the pole figure data, which distorted the texture analysis results reported in Lambda Report 5-8728. Because the pole figures and texture analysis results looked atypical, the client requested that Lambda recheck the analysis. Lambda confirmed and reported to the client in Lambda Report 5-8796 that the original results reported in 5-8728 were in error.

Describe cause. The specimen was not flat enough to align the specimen with the sample holder reference surface well enough to collect good pole figure data when the glass plate alignment procedure in 3P1016 is used. The greatest deviations from flatness occurred because the technician did not follow the directions for use of the contact cement required by procedure 3P1066. Epoxy was used to help hold the sample in place, which is not part of the procedure. Because the specimen was wavy, high spots on the sample were aligned with the sample mounting ring reference surface when a glass plate was used to position the specimen. The research engineer and technician did not recognize this positioning problem. Because most of the examined region on the sample was not positioned properly, the program Polfig did not find an appropriate two-theta angle for the (00.2) data collection and prompted for entry of an angle. The technician entered the angle listed in the technique file: it was at nearly the same angle as the peak he observed in a two theta scan of the specimen. Because the angle was not appropriate, measured intensities were too low at low tilt angles and too high at high tilt angles. The distortions were particularly large for the (00.2) pole figure. This caused distortion of the texture analysis. Two observations support these conclusions. First, when Polfig was run with a flatter specimen prepared from the same sample using only the contact cement according to its instructions as required by 3P1066, the program found the peak and the pole figure appeared to be normal. Second, when the original wavy specimen was run after aligning the flatter center of the specimen rather than raised portions with the reference surface, Polefig found the peak and generated pole figure data that appeared to be normal.

Describe Solution:

1) Flat specimens must be prepared and positioned correctly. Technicians must follow the procedures and instructions and be trained to prepare flat specimens.

2) The technicians and research engineers must be able to recognize if a specimen is not flat enough to obtain accurate pole figure data. Establishing a measurement or verifiable criteria for surface flatness should correct this situation. The current pole figure procedure 3P1016 requires that samples be flat within 0.002 inches. Due to the spring-like properties of the thinned tube samples, the current sample preparation method described in 3P1016 produces specimens that are not flat within 0.002 inches. Specimens appear to have raised areas whose heights vary by more than 0.002 inches, making appropriate sample positioning using the glass plate method in 3P1016 problematic. Currently, evaluation of specimen flatness is not performed in the lab.

Actions Taken or To Be Taken:

1) The client, GE Nuclear has been issued report 5-8796, which includes corrected data for 986985-01 and a description of the problem for this particular specimen.

2) Since its possible that this problem has occurred in the past and was not recognized, previous results of zircalloy tube analysis should be reviewed in order to determine if this problem has caused significant distortion of texture analysis results.

was notified data were reviewed.

3) ~~The client should be~~ notified ~~of the~~ possibility of pole figure data distortion due to lack of sample flatness and ~~positioning~~ so that they can assess ~~the significance of the problem~~. This is required by their PO per 10 CFR 21.

See attached list

4) 3P1066 and/ ~~or~~ 3P1016 should be revised to ensure production of flat specimens and verification that specimens are sufficiently flat and appropriately positioned for texture analysis. Directions for entry of appropriate peak positions when the automatic peak finding file fails should be addressed in the procedure. Technicians and engineers will be trained to ensure that they execute the procedures properly.

Already present

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Lambda Research QA Incident Report, 8/26/99, Attachment 1.

Description of Problem: Because the pole figures and texture analysis results looked atypical, the client, GE Nuclear requested that Lambda recheck the analysis of a specimen prepared from sample 986985-01 reported in Lambda Report 5-8728. Lambda reran the analysis on a freshly prepared specimen from the same sample and confirmed and reported to the client in Lambda Report 5-8796 that the original results reported in 5-8728 were in error. The original specimen from sample 986985-01 was not prepared flat enough for the analysis. This introduced errors in the pole figure data, which distorted the texture analysis results reported in Lambda Report 5-8728.

Describe cause. The specimen was not flat enough to align the specimen with the sample holder reference surface well enough to collect good pole figure data when the glass plate alignment procedure in 3P1016 is used. The greatest deviations from flatness occurred because the contact cement required by procedure 3P1066 was not used properly and the specimen would not adhere well to the backing. Epoxy was used to help hold the sample in place, which is not part of the procedure. Because the resulting specimen was wavy, high spots on the sample were aligned with the sample mounting ring reference surface when a glass plate was used to position the specimen. This positioning problem was not recognized. Because most of the examined region on the sample was not positioned properly, the program Polfig did not find an appropriate two-theta angle for the (00.2) data collection and prompted for entry of an angle. The technician entered the angle listed in the technique file: it was at nearly the same angle as the peak he observed in a two theta scan of the specimen. Because the angle was not appropriate, measured intensities were too low at low tilt angles and too high at high tilt angles. The distortions were particularly large for the (00.2) pole figure. This caused distortion of the texture analysis. Two observations support these conclusions. First, when Polfig was run with a flatter specimen prepared from the same sample using only the contact cement according to its instructions, the program found the peak and the pole figure appeared to be normal. Second, when the original wavy specimen was run after aligning the flatter center of the specimen rather than raised portions with the reference surface, Polefig found the peak and generated pole figure data that appeared to be normal.

Describe Solution:

1) Flat specimens must be prepared and positioned correctly. Technicians must follow the procedures and instructions for use of the glue and be trained to prepare flat specimens. The procedures should clearly describe the flatness requirement and use of the contact cement.

2) The technicians and research engineers must be able to recognize if a specimen is not flat enough to obtain accurate pole figure data. Establishing a measurement or verifiable criteria for surface flatness should correct this situation. The current pole figure procedure 3P1016 requires that samples be flat within 0.002 inches. Due to the spring-like properties of the thinned tube samples, the current sample preparation method described in 3P1016 produces specimens that are not flat within 0.002 inches. Specimens appear to have raised areas whose heights vary by more than 0.002 inches, making appropriate sample positioning using the glass plate method in 3P1016 problematic. Currently, evaluation of specimen flatness is not performed in the lab.

3) The automatic peak finding program and procedure must be fixed so that data is collected at the appropriate peak position. Currently, the peak finding program is not reliably finding the appropriate data collection angle and has caused distortions in the pole figure data. This problem becomes worse if the specimen is not flat. Its also recommended that the peak collection angle be actively reviewed by a technician or engineer prior to data collection.

Actions Taken or To Be Taken:

1) The client, GE Nuclear has been issued report 5-8796, which includes corrected data for 986985-01 and a description of the problem for this particular specimen.

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2) Since its possible that this problem has occurred in the past and was not recognized, previous results of zircalloy tube analysis should be reviewed in order to determine if this problem has caused significant distortion of texture analysis results.

3) The client should be notified of the possibility of pole figure data distortion due to lack of sample flatness and positioning and failure of the peak finding program so that they can assess the significance of the problem. This is required by their PO per 10 CFR 21.

4) 3P1066 and/ or 3P1016 should be revised to ensure production of flat specimens and verification that specimens are sufficiently flat and appropriately positioned for texture analysis. Directions for entry of appropriate peak positions when the automatic peak finding file fails should be addressed in the procedure. Technicians and engineers will be trained to ensure that they execute the procedures properly. The peak finding program and 3P1016 should be revised to ensure reported data was collected at the appropriate angles.

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