Measurement of Residual Stresses in Single Crystal Cubic Materials Using X-Ray Diffraction Technique
应用X射线衍射技术测量立方结构单晶材料的残余应力

Dr. Mohammed Belassel,
Proto Manufacturing Ltd., 2175 Solar Crescent, Oldcastle, Ontario, Canada.
E-mail: mbelassel@protoxrd.com   Web: www.protoxrd.com

Notice

Please note some of the designs, techniques and applications shown in this presentation are subject to copyright, patents granted or pending.

本文所应用的设计，技术及应用受版权,专利法的保护

Data is used with the permission of the respective owner(s) and may not be reproduced without permission.

本文采用数据已经授权, 未经许可,不得复制

Material Microstructures 材料微观结构

- Polycrystalline with coarse grain structure oriented (with preferred orientation)

多晶粗粒结构沿一定方向取向

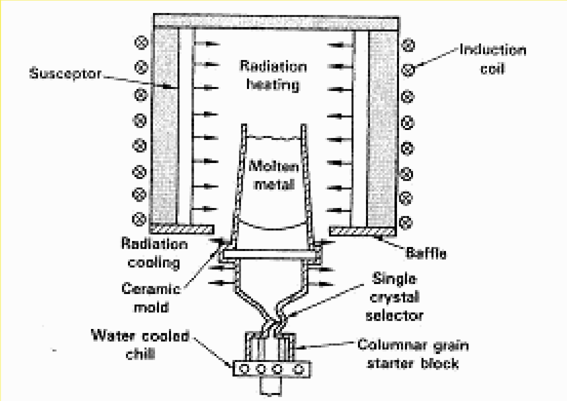
- Directional solidified grain structure

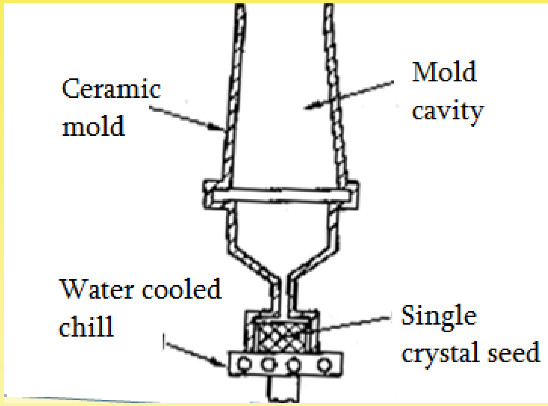
沿一定方向强化的晶粒结构

- Single Crystal structure

单晶结构

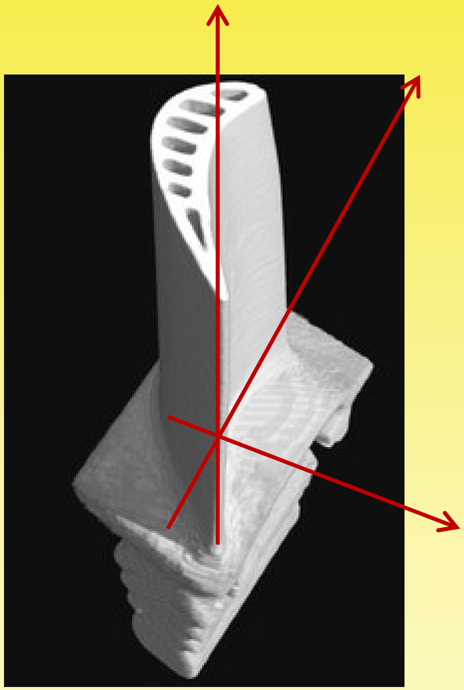
Fabrication of Material Components
材料部件的制造 Single Crystal Materials 单晶材料





Directional solidification Process to cast single Crystals and equiaxed grains
使用定向强化工艺从冷却铜版生产单晶**(**等轴晶粒**)**

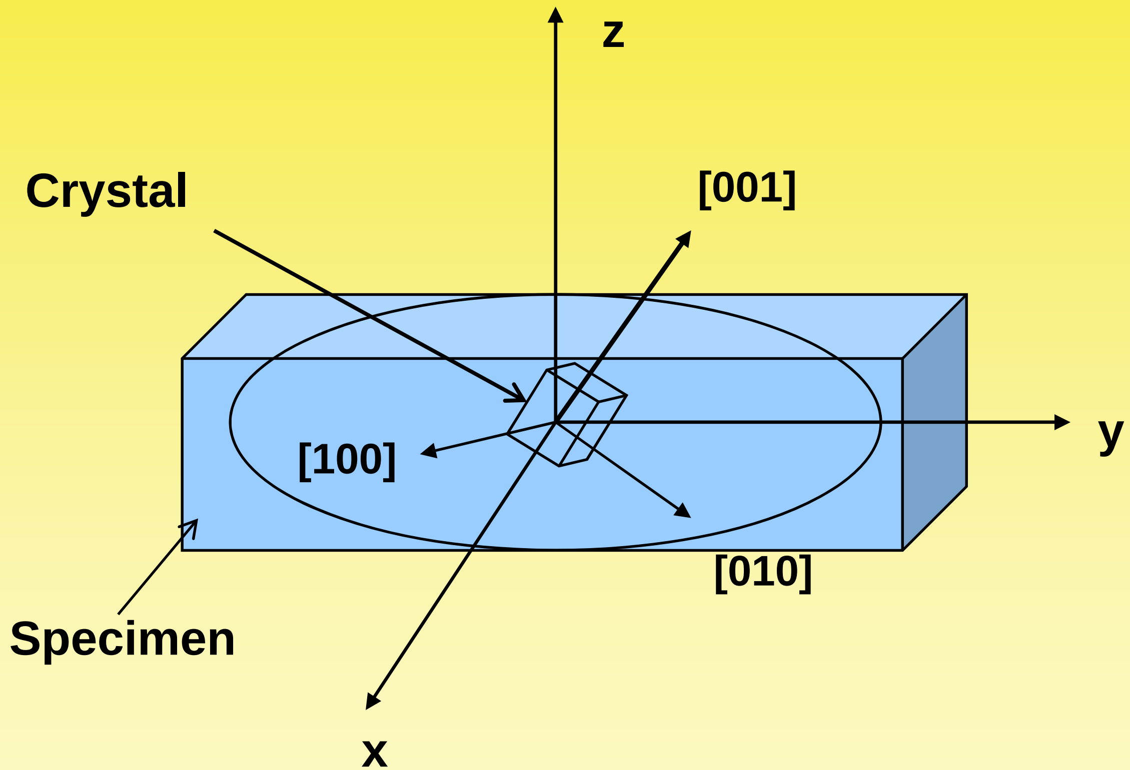
Fabrication of Material Components
材料部件的制造 Single Crystal Materials 单晶材料



Example of Turbine Blades

Stress Measurement Techniques 应力测量技术

Crystal Orientation (Reference definition)晶体方向（坐标系定义）

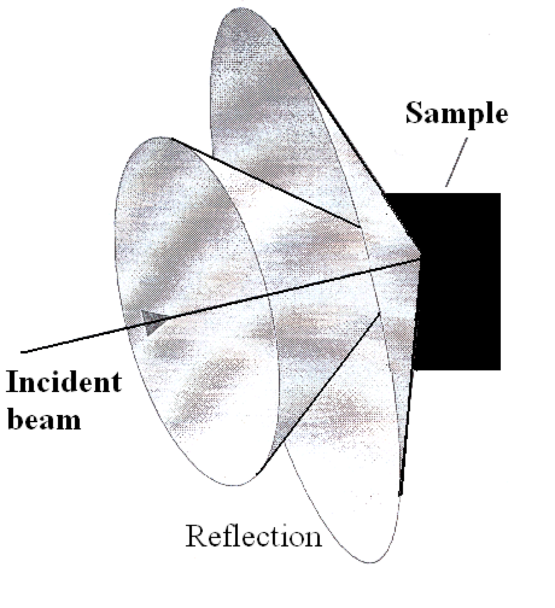


Transition form Crystal to specimen reference

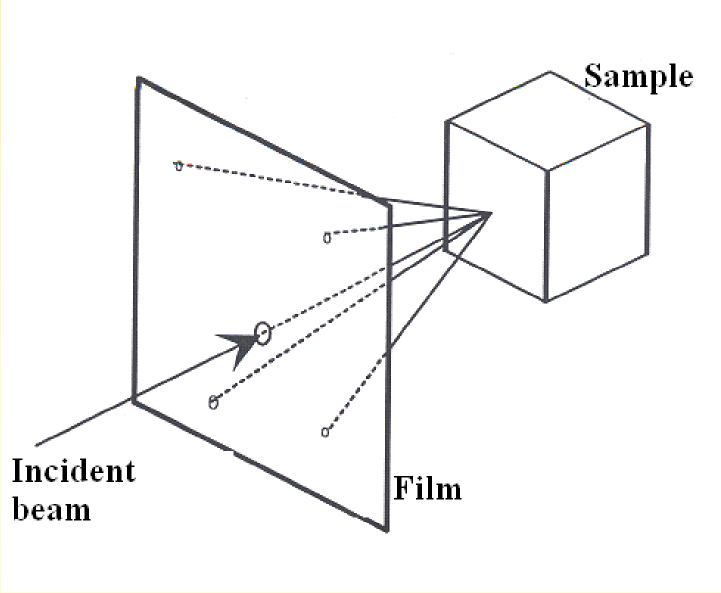
试样坐标系对晶体参照系的变换

Diffraction Techniques 衍射技术

- Polycrystalline Material and Single Crystal Materials 多晶材料和单晶材料



Debye Rings
德拜环



Discrete Poles (LAUE)
劳厄法

Mechanical Behaviour of Materials 材料的机械特性 Single Crystal materials 单晶材料

- Hooke's Law
  - Anisotropic materials 各向异性材料 $\sigma_{ij} = C_{ijkl}\varepsilon_{kl}$
  - 81 different components which reduces to 3 for cubic symmetry

    对立方体结构, 将81种不同的分量减少到只有三个分量

$$\sigma_{ij} = \begin{pmatrix} \sigma_{11} \\ \sigma_{22} \\ \sigma_{33} \\ \sigma_{23} \\ \sigma_{31} \\ \sigma_{12} \end{pmatrix}, C_{ijkl} = \begin{pmatrix} C_{11} & C_{12} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{11} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{12} & C_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & C_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & C_{44} \end{pmatrix}, \varepsilon_{ij} = \begin{pmatrix} \varepsilon_{11} \\ \varepsilon_{22} \\ \varepsilon_{33} \\ \varepsilon_{23} \\ \varepsilon_{31} \\ \varepsilon_{12} \end{pmatrix}$$

Mechanical Behaviour of Materials材料的机械特性 Single Crystal materials单晶材料

- Elastic behaviour 弹性特性
  - Elastic modulus in one direction 一个方向的弹性模量

$$\frac{1}{E^{[hkl]}} = S_{11} - 2\left((S_{11} - S_{12}) - \frac{1}{2}S_{44}\right).\Gamma^{[hkl]})$$

$$\Gamma^{[hkl]} = \frac{h^2k^2 + k^2l^2 + l^2h^2}{\left(h^2 + k^2 + l^2\right)^2}$$

  - $\Gamma^{[100]}=0$, $\Gamma^{[110]}=1/2$, $\Gamma^{[111]}=1/3$
  - For isotropic matrerials $\left((S_{11} - S_{12}) - \frac{1}{2}S_{44}\right) = 0$

Stress Measurement Techniques 应力测量技术 Single Crystal Material 单晶材料

- Steps for measuring stresses in a single crystal
  测量单晶应力的步骤
  - Determine the crystal orientation using Pole figure technique or LAUE technique, scan in ϕ and ψ directions using low multiplicity (hkl) plane
    利用极图或LAUE技术,确定晶体的方向, 用低阶(hkl)平面在ϕ and ψ方向扫描
  - Select at least 3 poles or more to determine an accurate crystal orientation
    选择至少 3 个极点或更多来确定精确的晶体方向
  - Index the poles (hkl)
    检索极点（hkl)

Stress Measurement Techniques 应力测量技术 Single Crystal Material 单晶材料

- Calculate the orientation matrix
  计算定向矩阵
- Calculate the poles positions for stress measurements, new (hkl) planes
  计算应力测量的极点的位置，新的hkl平面
- Scan in half space around the pole and determine accurately Bragg angle of the peak
  对极点进行上半空间空间扫描，决定精确的峰位的Bragg角
- Using the elasticity theory, calculate the stress tensor.
  应用弹性理论，计算应力张量

X-Ray Diffraction Equipment X射线衍射仪 Configuration 配置

The equipment should be equipped with the following: 仪器应该有如下配置

- Motorized ψ rotation, $0 \le \psi \le 60$
  自动化ψ角旋转范围，
- Motorized ϕ rotation $0 \le \phi \le 360$
  自动化ϕ 旋转范围，
- Linear detector or 2D detector
  线性探测器或二维探测器

Proto LXRD System

X-Ray Diffraction Equipment X射线衍射仪 Configuration 配置

- X-Y stage for easy positioning
  自动化的X-Y平台灵活定位
- High power X-ray tube
  大功率X射线管
- Safety enclosure
  带安全锁的机柜
- Control and analytical software
  控制和分析软件

Stress Measurement Techniques 应力测量技术Alignment verification, ASTM E915 校准验证

Free stress powder: *316L* *无应力316L粉*

X-Ray tube: *Mn* *靶材*

Plane (hkl): *311* *晶面*

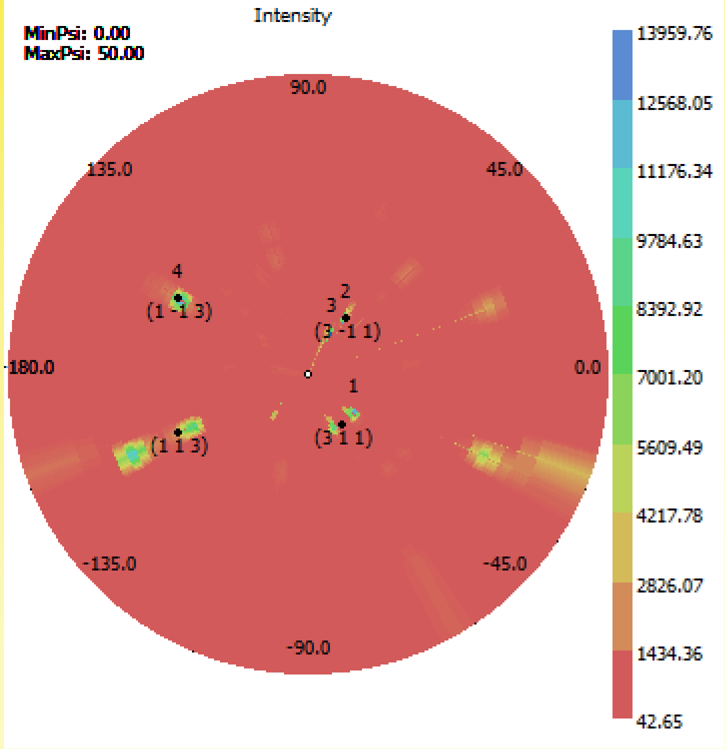
Bragg angle, 2θ = *152°* *布拉格角*

XEC = $7.18.10^{-6}$ MPa^{-1}

$\sigma = 1.7 \pm 6.7$ Mpa

$\tau = 6.2 \pm 3.4$ MPa

Stress Measurement Techniques应力测量技术Crystal Orientation (Pole figure technique)晶体方向极图法

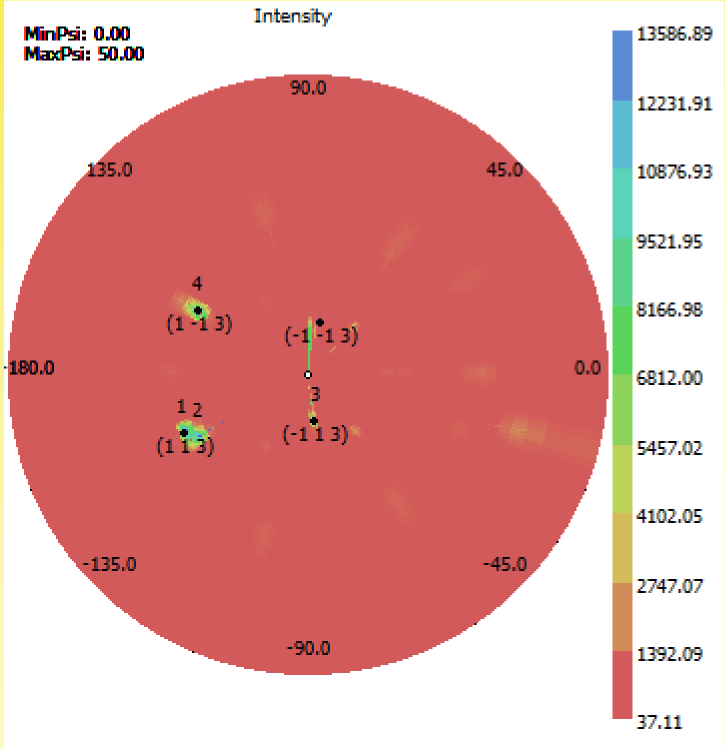


Sample 1: 样本一
X-Ray tube: *Mn*
Plane (hkl): *311*
Bragg angle, 2θ = *155°*

Pole figure collected on a Nickel Base Single crystal

镍基单晶中采集的的极图

Stress Measurement Techniques应力测量技术Crystal Orientation (Pole figure technique)晶体方向极图法



Sample 2: 样本二

X-Ray tube: *Mn*

Plane (hkl): *311*

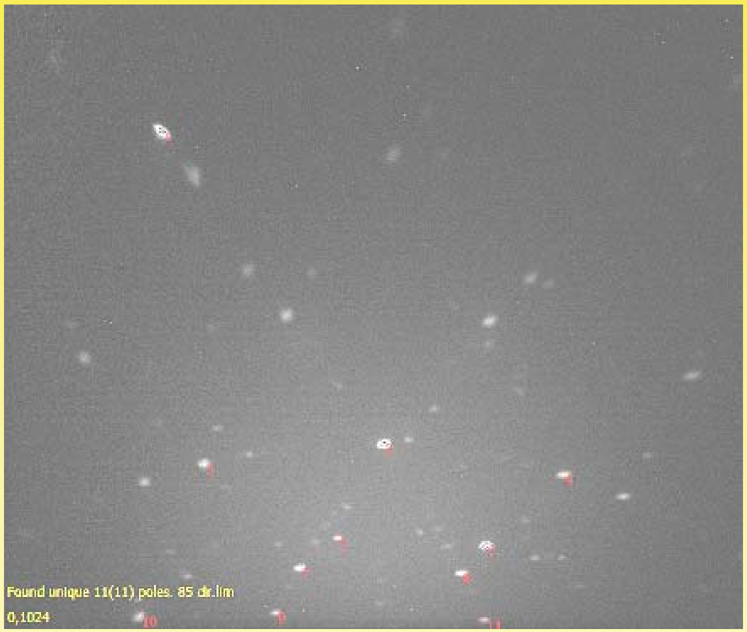
Bragg angle, $2\theta = 155^{\circ}$

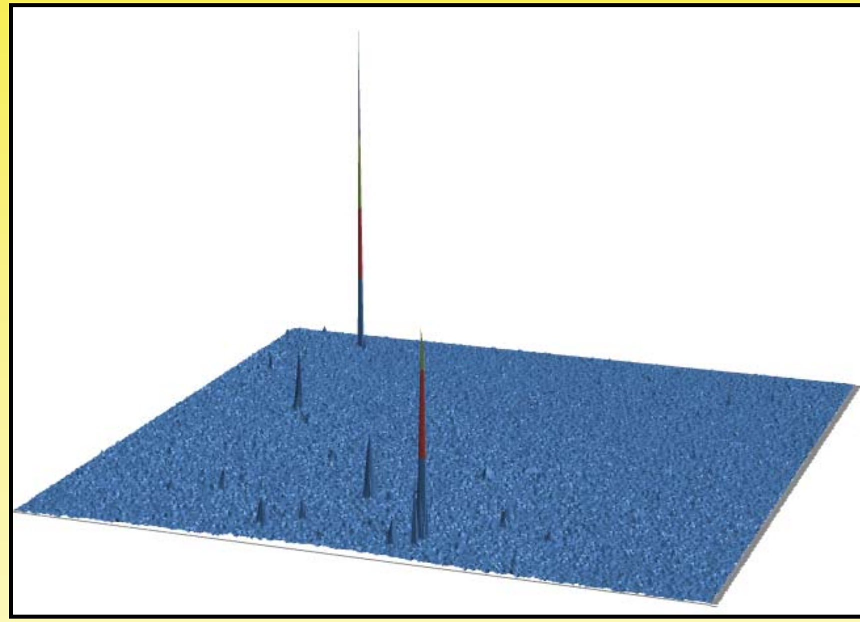
Pole figure collected on a Nickel Base Single crystal

镍基单晶中采集的的极图

Stress Measurement Techniques应力测量技术

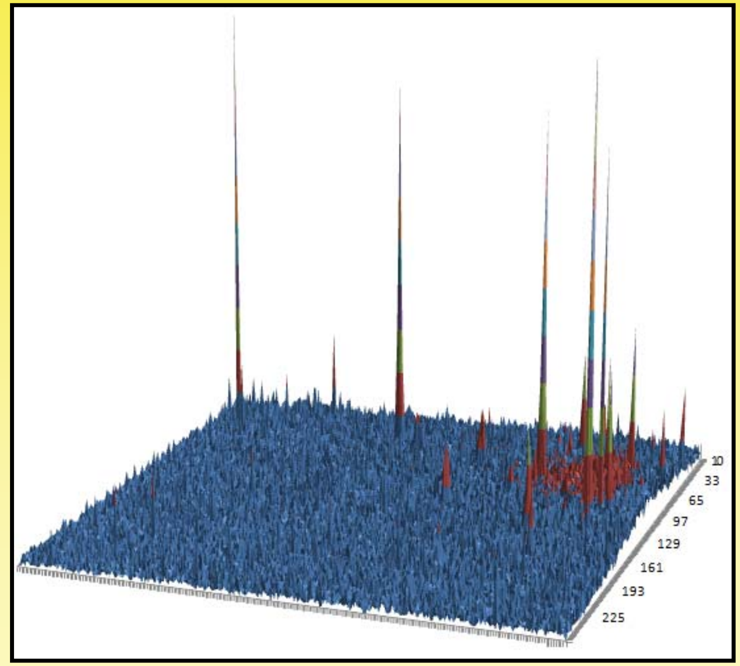
Crystal orientation (LAUE technique)晶体方向（劳厄法）





- **Digital LAUE photograph and a 3D LAUE data, Sample 1**
  劳厄数码照像与三维劳厄数据，　样本一

Stress Measurement Techniques应力测量技术Crystal orientation (LAUE technique)晶体方向（劳厄法）



- **Digital LAUE photograph and a 3D LAUE data, Sample 2**
  劳厄数码照像与三维劳厄数据，　样本二

Stress Measurement Techniques应力测量技术
Crystal Orientation Pole figure versus LAUE techniques
晶体定向，极图法对劳厄法

- Single crystal orientation accuracy can be affected by the following:　影响单晶定向精度的因素
  1. Intensity of peaks　峰强度
  2. Poles overlap　极点重叠
  3. Mosaicity in the crystal　晶体中的马赛克
  4. Accuracy of poles position determination　极点定位精度
  - Solution　解决方案

1. Select low multiplicity peak for orientation选择低阶ＨＫＬ平面峰定向
2. High resolution detector　高分辨探测器
3. High power for high intensity peaks　大功率得到强峰

Stress Measurement Techniques应力测量技术 Deployment 实施

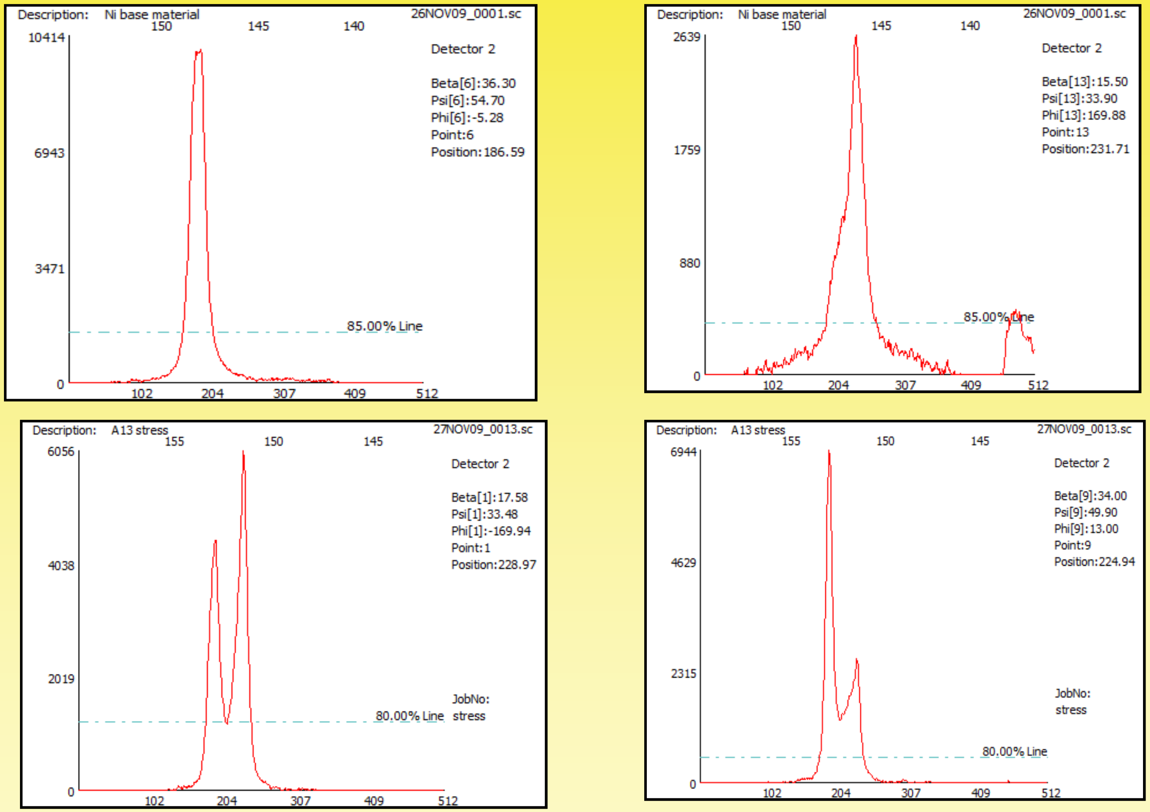
- Use the single crystal orientation result to predict the position of the new poles, usually higher multiplicity
  利用确定的单晶的方向预测新极点的位置，通常是较高阶的H K L平面
- Calculate the coordinates of the new poles (ψ,ϕ)
  计算新极点的坐标(ψ,ϕ)
- Scan in space around the theoretical pole position to find the pole maximum position
  在理论新极点位置附近扫描发现极点峰位

Stress Measurement Techniques应力测量技术 Deployment 实施

- Collect a peak at the maximum position in (ψ,ϕ) and calculate the peak position in 2θ
  在极点峰位处(ψ,ϕ) 采集衍射峰，计算峰位的2θ
- Calculate strain
  计算应变
- Using the elasticity theory, calculate the stress tensor.
  应用弹性理论，计算应力张量

Stress Measurement Techniques应力测量技术

Results 结果

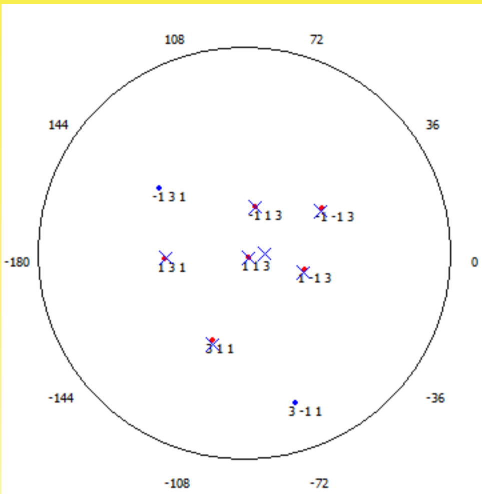


Example of x-ray diffraction peaks collected on a single crystal using Mo X-Ray tube

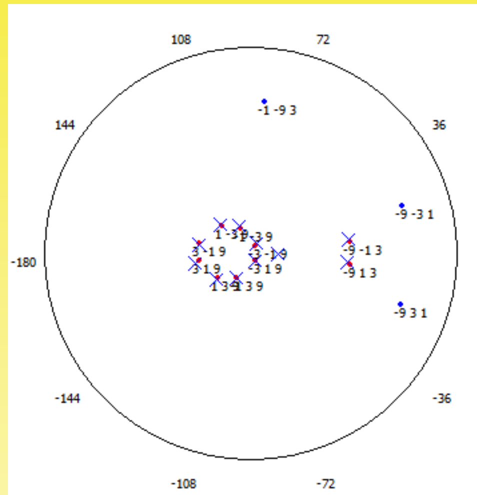
实例：使用ＭＯ靶Ｘ光管采集的单晶Ｘ光衍射峰

Stress Measurement Techniques应力测量技术

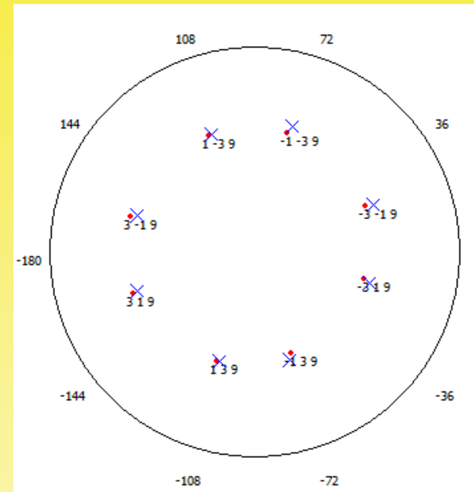
Results 结果



Mn target



Mo target



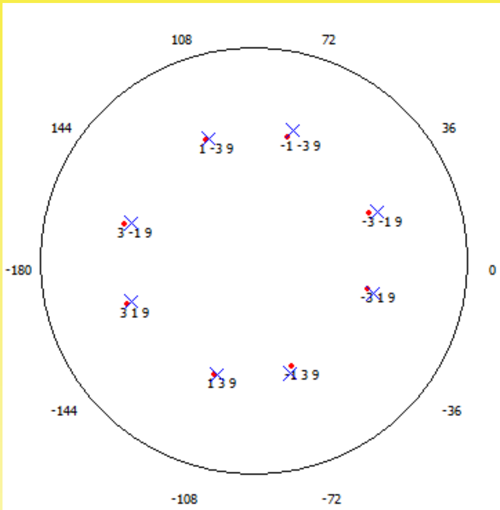
Mo target

Example of poles layout collected on a single crystal

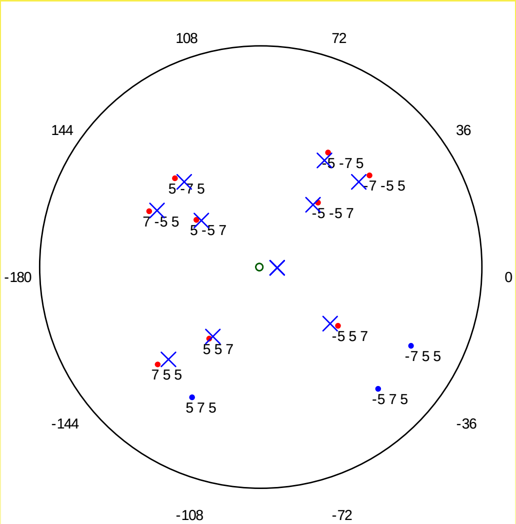
实例：从单晶中采集的极点分布图

Stress Measurement Techniques应力测量技术

Results　结果



Mo target



Mo target

Example of poles layout collected on a single crystal

实例：从单晶中采集的极点分布图

Stress Measurement Techniques应力测量技术
Results 结果

Single Crystal

JobNo:
PartNo:
Description: Ni base material

Date: :
Time:
Operator
Elapsed Time:

Stress Tensor [MPa]			**Error [MPa]**		
-272.85	-73.90	81.16	0.00	0.00	0.00
	-219.31	97.11		0.00	0.00
		-0.00			0.00

	Calculated		Measured			
Plane	Phi	Psi	Phi	Psi	dspacing	
(1 3 1)	-175.76	47.21	-176.26	46.71	1.082391	V
(3 1 1)	-109.50	52.39	-109.00	53.39	1.082026	V
(3 -1 1)	-71.13	67.50	0.00	13.60	N/A	
(1 1 3)	-46.48	4.65	-47.48	5.15	1.085376	V
(1 -1 3)	-16.01	39.20	-17.51	39.20	1.084397	V
(-1 -1 3)	29.96	51.11	28.96	50.61	1.083755	V
(-1 1 3)	75.83	31.25	76.33	30.25	1.085336	V
(-1 3 1)	142.62	57.10	0.00	13.60	N/A	

Example of Residual Stress results on a single crystal, shot peened subsurface

实例：单晶残余应力的计算结果

Summary

- Single crystal and Directionally Solidified materials can be characterized for both Crystal Orientation and Residual Stress
  单晶和定向强化材料能够量化分析其晶体方向和残余应力
- Measurements were successfully applied in both lab and factory settings using Proto XRD equipment
  应用Proto公司的ＸＲＤ设备实验室设置和工厂设置成功地进行了有效的分析和测量
- Future development will include fast speed systems for LAUE and stress
  未来的开发主要包括高速系统