# Micro area X-ray stress measurement system
# AutoMATE II

## 1. Introduction

We have improved the conventional micro area stress measurement system (AutoMATE) by replacing the gas flowing detector Position Sensitive Proportional Counter (PSPC) with a semiconductor detector, the D/teX Ultra 1000. This new AutoMATE II provides the user with new functions, high sensitivity, high counting rate and good energy resolution. Maintenance of the AutoMATE II is much easier than that of AutoMATE because there is no longer the need to flow PR gas. The higher sensitivity of the D/teX Ultra 1000 enables the user to perform micro-area stress measurements in less time. The high counting rate capability of the D/teX Ultra 1000 means that the stress measurement of crystalline materials having coarse grains or textures can be performed easily.

## 2. Advantages of the D/teX Ultra 1000 1D detector

### 2.1. High Sensitivity

The D/teX Ultra 1000 is a Silicon Strip Detector (SSD), a semiconductor detector with high sensitivity and high counting rate. Figure 1 shows D/teX Ultra 1000 installed on the AutoMATE II system. In Fig. 2, two diffraction profiles for the 211 reflection of $\alpha$Fe observed with the PSPC and the D/teX Ultra 1000, respectively, are shown. These profiles were measured with a collimator of 1 mm. The intensity at the peak top of the profile observed with the D/teX Ultra 1000 is about 1.7 times as large as that with the conventional PSPC.

### 2.2. High Counting Rate

D/teX Ultra 1000 can count up to $10^6$ counts per second (cps) per strip. The global counting rate over the total 1024 strips is in excess of $10^9$ cps. The design of the D/teX Ultra 1000 provides for high sensitivity as well. Therefore, the D/teX Ultra 1000 can detect high intensity reflections from a sample having coarse grains or textures without dead time corrections. Utilizing a function of the high counting rate, the D/teX Ultra 1000 is applicable to residual stress measurements processing diffraction profiles from those samples with coarse grains, such as welded parts, electromagnetic steels, aluminum parts, etc., without regard to the effect of dead time. In Fig. 3, two diffraction profiles observed with the PSPC and the D/teX Ultra 1000, respectively, are shown, which belong to the 211 reflection of $\alpha$Fe. The

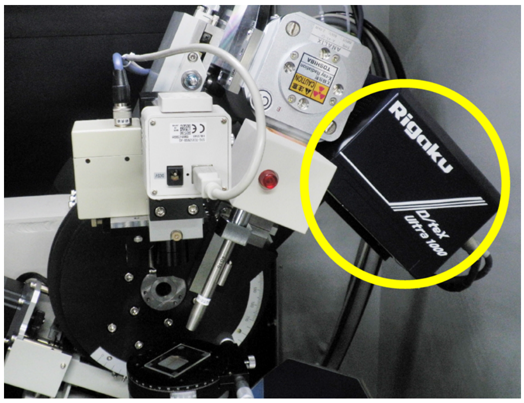
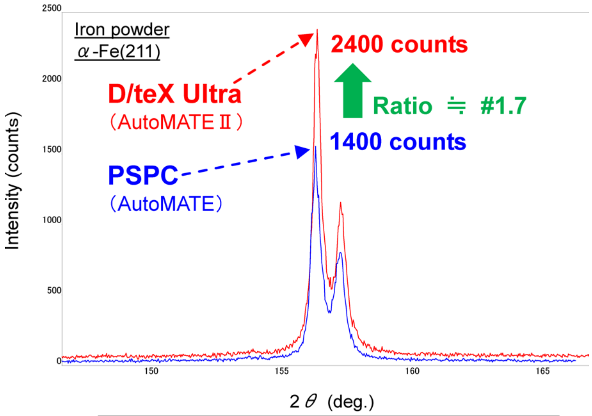
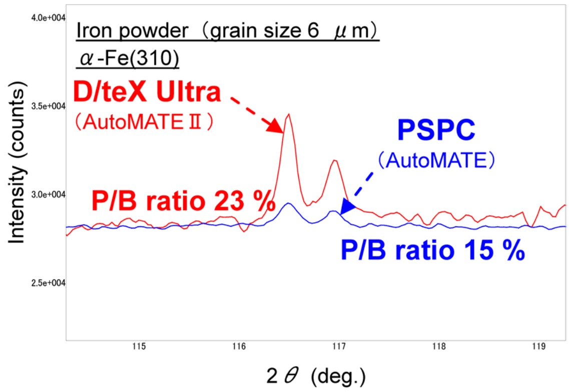



**Fig. 1.** Semiconductor one-dimensional detector, D/teX Ultra 1000.



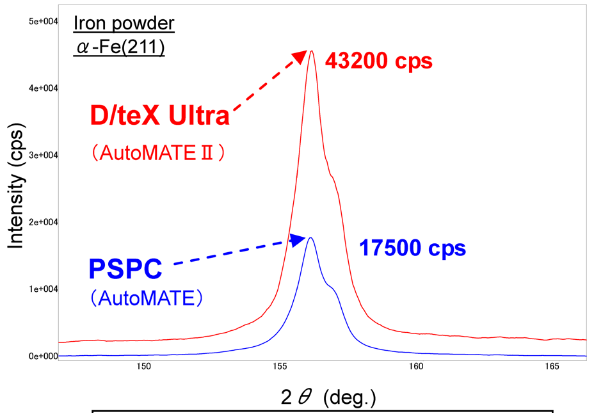

<Measurement Parameters>
- X-ray source: Cr $K\alpha$
- Tube voltage-Tube current: 40 kV-40 mA
- Collimator diameter: 1 mm
- Measurement time: 20 sec.
- Reflection plane: α-Fe(211)
- Diffraction angle without strain: $2\theta_0$=156.08°
- Step width: 0.03°

**Fig. 2.** Diffraction peaks of the 211 reflection observed with a new and an old AutoMATE II.




<Measurement Parameters>
- X-ray source: Cu$K\alpha$
- Tube voltage-Tube current: 40 kV-40 mA
- Collimator diameter: 1 mm
- Measurement time: 400 sec.
- Reflection plane: α-Fe(310)
- Diffraction angle without strain: $2\theta_0$=116.38°
- Step width: 0.03°

**Fig. 4.** Energy resolution of D/teX Ultra 1000 and PSPC.




<Measurement Parameters>
- X-ray source: Cr$K\alpha$
- Collimator diameter: 4 mm
- Measurement time: 20 sec.
- Reflection plane: α-Fe(211)
- Diffraction angle without strain: $2\theta_0$=156.08°
- Step width: 0.05°

**Fig. 3.** Maximum count rates of D/teX Ultra 1000 and PSPC.

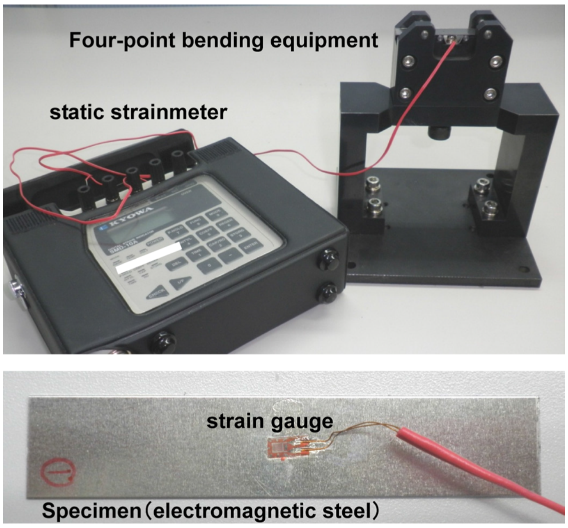



**Fig. 5.** Four point bending test of an electromagnetic steel.

counting rate (43200 cps) at the peak top of the profile observed with the D/teX Ultra 1000 is nearly 2.5 times as large as the counting rate (17500 cps) for the PSPC.

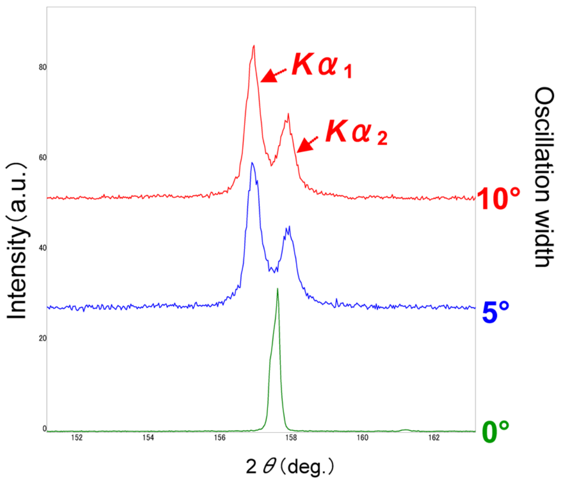
## 2.3. High Energy Resolution

The energy resolution capability of D/teX Ultra 1000 enhances the peak-to-background (P/B) ratio of observed reflections, which is much improved compared to that for the PSPC. Thus, X-rays from sample fluorescence, which are emitted from iron-based metals with Cu X-ray source and often prevent detecting main reflections, are efficiently reduced.

In Fig. 4, two diffraction profiles observed with the PSPC and the D/teX Ultra 1000, respectively, are shown, belonging to the 310 reflection of αFe. The P/B ratio (23%) of the profile observed with the D/teX Ultra 1000 is nearly 1.5 times as large as the P/B ratio (15%) for the PSPC.

## 3. Application (Stress Measurement with Oscillation)

Electromagnetic steel is an iron-based metal, which derives its magnetic properties from iron. Addition of silicon up to a few percent improves the efficiency by which electric energy is converted to magnetic field strength. It is well known that for electromagnetic steel, the core loss (power loss) is reduced when the amount of silicon is increased up to a threshold. At the threshold, cracks will be easily induced in specimens because




**<Measurement Parameters>**
- X-ray source: Cr$K\alpha$
- Tube voltage-Tube current: 40 kV-40 mA
- Collimator diameter: 1 mm
- Measurement time: 1 min. to 2 min.
- Reflection plane: $\alpha$-Fe(211)
- Diffraction angle without strain: $2\theta_0$=156.08°
- Step width: 0.03°

**Fig. 6.** Effects of oscillation in electromagnetic steels (collimator diameter 1 mm, $\phi$=0°)

of the constituent coarse grains grown relative to the amount of silicon.

Figure 5 shows an electromagnetic steel specimen loaded with a four-point bending equipment connected to a static strainmeter, where the measurement system is shown with a close-up picture of the sample with a strain gauge in place. In Fig. 6, three diffraction profiles of the 211 reflection of $\alpha$Fe in an electromagnetic steel specimen with the coarse grains of 100 $\mu$m were observed by changing the oscillation widths from 0° to 10° by 5° step, respectively, using the D/teX Ultra 1000 and a collimator of 1 mm.

For no oscillation (0°), the diffraction profile was not observed at the correct diffraction angle. However, for the oscillation widths of 5° and 10°, those observed diffraction profiles contained two peaks separated by $K\alpha_1$ and $K\alpha_2$ lines.

In Table 1, residual stresses observed with oscillation widths of 5° and 10° in the two directions of the long and short sides are listed, respectively. The residual stresses obtained are good agreement with each other without reference to oscillation widths.

**Table 1.** Results of the residual stress measurements in the loaded electromagnetic steel with the constituent grains of 100 $\mu$m.

| Collimator diameter | Oscillation width | Stress±Confidence limit (MPa) | |
|---|---|---|---|
| | | Long side | Short side |
| $\phi$1 mm | ±10° | 247.44±9.91 | −33.68±28.40 |
| | ±5° | 248.35±15.11 | −41.12±31.73 |
| | 0° | — | — |