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Report

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DEPC-MPE 035

INVESTIGATION OF AN ELASTIC MODULUS REFERENCE MATERIAL

R MORRELL

NOT RESTRICTED

January 2007

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R Morrell
Division of Engineering and Process Control

ABSTRACT

A commercial single crystal silicon material has been investigated for use as an elastic modulus reference material using the impact excitation method. The results show good consistency between the three samples tested, and a very close to the literature data for silicon. The average flexure-derived data supplied with the material are also close to the results in the current study, but the scatter/uncertainty of the former is much greater than that achieved using the impact excitation method.

This work has enabled indirect validation of the impact excitation system as well as providing confidence in the use of this material for other purposes, such as beam flexure and dynamic mechanical analysis (DMA).

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Approved on behalf of the Managing Director, NPL,
by Dr M G Gee, Knowledge Leader, Materials Performance Team
authorised by Director, Engineering and Process Control Division

CONTENTS

1. INTRODUCTION

The accurate measurement of the elastic properties of materials is frequently demanded by industry because of their importance in predicted design, especially in finite element analyses. There are a number of ways in which these properties can be derived, including:

- Quasistatic tensile tests
- Quasistatic flexural tests
- Dynamic rod, beam, disc or plate resonance
- Dynamic rod, beam, disc or plate impact excitation (natural frequency)
- Ultrasonic velocity measurements

The tensile testing method relies on extensometry or strain gauges to measure strain, and while widely used for metals, the method is critically reliant on correct test-piece alignment to avoid bending. If there is non-linearity of behaviour, or micro-creep involved, this is the method of choice for quasistatic behaviour. because the alternative, flexural testing, gives indeterminate results unless the material remains elastic. Using appropriate strain gauges, Poisson's ratio can also be determined. Typically, Young's modulus can be determined to an accuracy of 5% by this method.

Flexural testing is simpler, especially for brittle materials, since deflections are larger than in the tensile case. For long, thin beams, tested, for example in a conventional universal mechanical testing machine, an accuracy of a Young's modulus determination can be good as 2%, although more typically 5% is achieved on small test-pieces. If strain gauging rather than deflection measurement is used, then again, Poisson's ratio can be determined.

The dynamic resonance and impact excitation methods rely on the natural modes of vibration of a test-piece when excited respectively, continuously or by discrete impact events. In contrast with the mechanical methods above, there is no strain or displacement measurement to be made, just a frequency, and generally this can be measured much more accurately. Increasingly, these methods are being employed because they are quick and simple, and commercial systems are available. Assuming that frequencies are correctly and accurately determined, the principal source of error is in the shape precision of the test sample and the accuracy of dimensional measurement, and results to better than 1% uncertainty are thought to achievable.

The ultrasonic method relies on determining the time of flight of an ultrasonic pulse between the parallel opposing faces of the test material, and thus the principal source of uncertainty is in the time-base of the electronic system recording the measurement and the sample dimension measurements.

Many of these issues have been reviewed in an NPL Good Practice Guide [1].

With particular regard to the frequency-based dynamic modulus measurement methods, to provide traceability of measurement of elastic properties requires that traceability is required for the following measurements that contribute to calculations:

- Mass – generally not a problem with modern balances to achieve 100 µg or better accuracy

- Dimensions – digital micrometers for dimensions up to 25 mm are typically accurate to better than ±0.002 mm, and digital callipers for dimensions up to 200 mm are typically accurate to ± 0.02 mm; more crucially it is the accuracy of shape and quality of the surface finish of the test-piece that needs to be taken into account
- Density – may need to be determined directly, *e.g.* by the Archimedes method, but often is substituted by mass and dimensions in computational software; direct measurements typically accurate to 0.5% or better
- Frequency – if frequency is determined directly by counting, then it can be calibrated directly from a frequency source and a calibrated counter, to a high precision, typically better than 1 part in 10^5; however, if it is determined indirectly, *e.g.* through a fast fourier transform analysis of a response signal, the accuracy may depend on the timebase within the instrument (e.g. a computer), and it becomes difficult to develop the traceability. Earlier work at NPL studied the possibilities of traceably calibrating the "Grindosonic" instrument[1] [2] using a frequency source, a traceably calibrated frequency counter and a loudspeaker with volume control to create a rapidly decaying sound pulse to trigger the instrument correctly.

An alternative approach is to use a reference material, but these are not widely available. Recently, some 100 x 10 x 1 mm single crystal silicon beams of <100> orientation with certified Young's modulus were supplied by the silicon wafer manufacturer PB-Technik AG, Zollikon, Switzerland via Mettler-Toledo (Switzerland and UK) as a potential reference material for DMA measurements. According to the certificate (Annex A), these had been directly calibrated using three-point flexure over a 90 mm span at a rate of 1 Hz to yield a mean (axial) 'typical' value of 128 GPa with 'minimum' of 120 GPa and a 'maximum' of 135 GPa, compared with a 'theoretical' value of 130.2 GPa [3].

This report cover the measurement of elastic properties of the three acquired samples using the impact excitation method on commercial equipment manufactured by IMCE [2] with a view to their use as reference samples.

2. TEST SAMPLES

The three samples were packed together and not separately identified. The 'certificate' identified them originating from a single crystal ingot of orientation only 12' 8" of arc off the <001> axis, but there is no confirmation that individually the samples have the same orientation as the ingot. This does not represent too much of a problem, however, since the effect of orientation angle on Young's modulus *E* for a cubic material is small up to several degrees off axis through the expression [4]:

$$E\ (\theta,\varphi) = (S_{11} - 2SJ)^{-1} \tag{1}$$

where: $S = S_{11} - S_{12} - S_{44}/2$ and S_{ij} are the single-crystal tensor compliances

and $J = \sin^2\theta \cdot \cos^2\theta + \sin^4\theta \cdot (1 - \cos 4\varphi)/8$

where θ is the azimuthal angle away from the <001> axis, and
ϕ is the rotation angle away from the (011) plane

[1] "Grindosonic", manufactured by Lemmens Elektronika BV, Leuven, Belgium; used as a QA tool in industry for determination of fundamental vibration frequencies.
[2] "HTVP1250" , manufactured by IMCE, Leuven, Belgium

For the record, details of the mass and dimensions of the samples are shown in Table 1. Width and thickness measurements were made at both ends and at the centre, and the average values taken.

Table 1 – Mass and dimensions of the three test samples

Sample	Mass*, g	Width**, mm			Thickness**, mm			Length***, mm
		End 1	Centre	End 2	End 1	Centre	End 2	
#1	2.3548	10.016	10.007	10.046	1.012	1.009	1.008	100.05
#2	2.3584	10.051	10.028	10.031	1.010	1.011	1.011	100.01
#3	2.3558	10.073	10.020	10.048	1.006	1.008	1.008	100.04

* Electronic balance, Mettler 240, UKAS calibrated and certified
** Digital micrometer, checked against certified gauge blocks
*** Digital callipers, checked against certified gauge blocks

3. MODULUS MEASUREMENTS USING IMPACT EXCITATION

The IMCE HTVP1250 impact excitation apparatus control computer and microphone sensor were used with the room temperature test-piece support frame. This consisted of an aluminium frame with parallel spring-tensioned nylon strings to act as test-piece supports. For flexural and longitudinal modes of vibration, the wires were set at the fundamental mode nodal lines of the test-piece, approximately 0.223 of the length of the test-piece from each end (Figure 1). For the torsional mode of vibration, the two strings were placed close together and the test items balanced on them at the centre. A microphone was set in a clamp stand and positioned over one end of the sample (flexural and torsional modes) or at one end in line with the length the sample (longitudinal mode).

The IMCE ambient temperature programme was employed. The parameters in Table 1 were input in order to directly compute the elastic moduli within the IMCE software.

The fast fourier transform (FFT) natural frequency spectra for sample #1 when supported for the flexural mode and struck at the centre and at the end are shown in Figure 2. When struck at the centre, the modes of vibration that have antinodes at the strike point (and nodes at or close to the support wires) dominate, whereas when it is struck near the end, all modes are excited. In addition, when struck at the end to one side (*i.e.* on the corner), torsional modes are also excited. The mode number of the peaks in the FFT can be identified by their ratio with the fundamental frequency, and through this the torsional modes (T1, T2, *etc.*) can be isolated from the flexural modes (F1, F2, *etc.*). This procedure acts as a check on the correct identification [1].

Table 2 shows the actual frequencies determined for sample #1 and the frequency ratios it can be seen that there is a close match with the theoretical values determined by numerical computation using the equations derived by Huang [5].

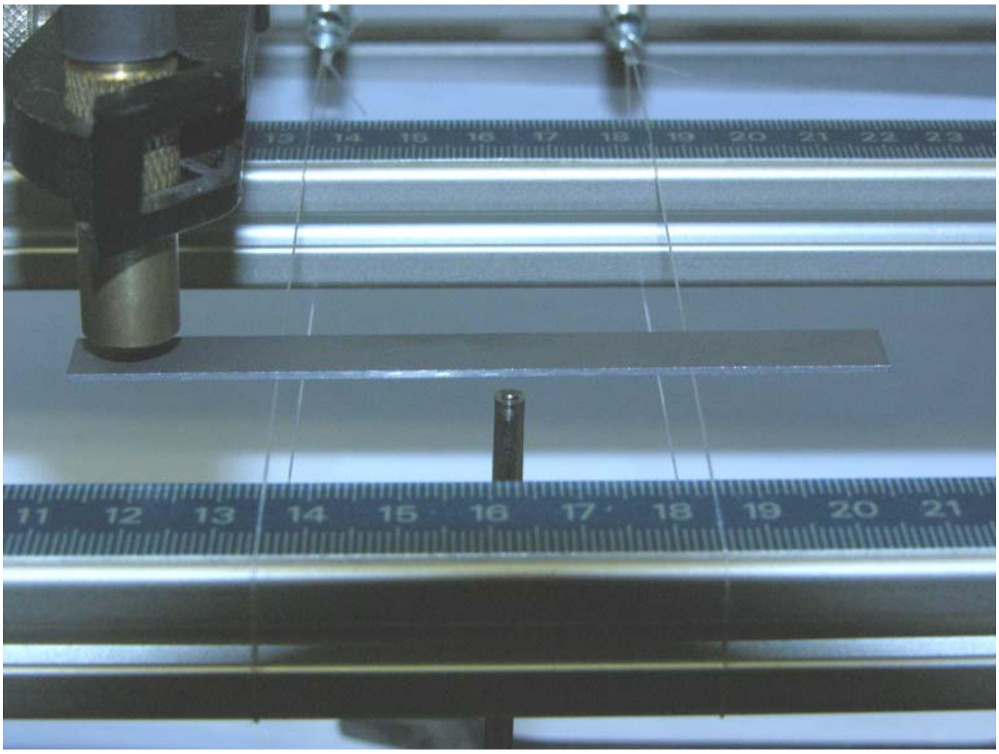

Figure 1: Silicon test-piece set up on two parallel nylon strings for central impact with microphone detection at one end.

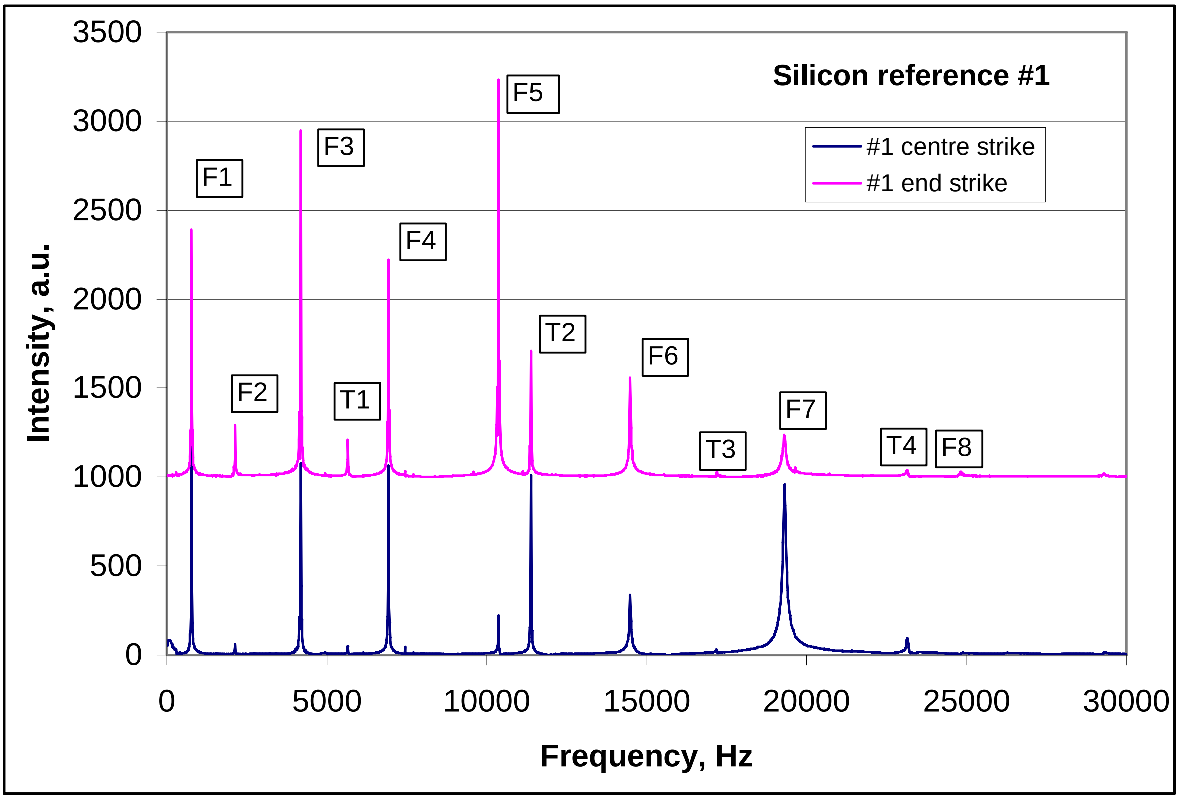

Figure 2: Fast fourier transform frequency spectra for sample #1 when struck in the through-thickness flexural mode at the centre and at the end.

Table 2: Modal frequency ratios for sample #1

	Vibration mode							
Flexural modes	***F1***	***F2***	***F3***	***F4***	***F5***	***F6***	***F7***	***F8***
Frequency, Hz	772	2138	4187	6930	10362	14492	19312	24822
Experimental frequency ratio *Fn/F1*	1	2.769	5.424	8.977	13.422	18.772	25.016	32.153
Theoretical ratio, *Fn/F1*	1	2.757	5.404	8.933	13.347	18.646	24.836	31.925
Torsional modes	***T1***	***T2***	***T3***	***T4***				
Frequency, Hz	5660	11385	17333	23144				
Experimental frequency ratio *Tn/T1*	1	2.011	3.062	4.089				
Theoretical ratio, *Tn/T1*	1	2	3	4				

In addition to the flexural experiments described above, the torsional mode vibrations were preferentially excited using central support and striking on one corner, detecting over the opposite corner. The longitudinal mode vibrations were obtained by striking on the end of the sample. Table 3 summarises the fundamental mode frequencies for flexure, torsion and longitudinal for all three samples. These values were consistent to within 1 Hz on repeated striking.

Table 3: Fundamental mode frequencies for all samples

	Fundamental mode frequencies, Hz		
	Flexural	**Torsional**	**Longitudinal**
Sample 1	772.0	5662	37283
Sample 2	773.0	5664	37301
Sample 3	772.5	5664	37294

The values of the elastic moduli determined from these frequencies in the different support and strike modes are summarised in Table 4. Five strikes were used in each mode and the average and standard deviations computed. The IMCE software requires the input of Poisson's ratio value for evaluating both longitudinal and flexural modes, which it does on the basis that the test sample is an isotropic material. Neither result for Young's modulus is normally particularly sensitive to the value employed. However, for a single crystal material the procedure is not so clear, as will be discussed below. The input value employed for these calculations was therefore the software default value of 0.25. In the case where both flexural and torsional fundamental modes can simultaneously be identified by the measurement system, Poisson’s ratio is computed independently of an input value, assuming the usual relationship between E and G for an isotropic material.

It can be seen that the scatter in the experimental results for an individual sample is small, while the differences between samples is rather larger. This latter arises primarily from the uncertainties in the input data for dimensions and the assumption that the shape of the test-piece is an ideal cuboid. It should also be noted that in the case of longitudinal measurements, there is uncertainty in the extant relationship between frequency and modulus [1] as well as a greater reliance on the input value of Poisson’s ratio.

4. COMPARISON OF TEST RESULTS WITH SINGLE CRYSTAL DATA

Data from Cunningham [6] of the US Bureau of Mines for the stiffness tensor elements for silicon are:

C_{11}	165.7 GPa
C_{12}	63.9 GPa
C_{44}	79.57 GPa

From equation (1) Young’s modulus in the <001> direction is given by:

$$E = 1/S_{11} = \frac{(C_{11} - C_{12})(C_{11} + 2C_{12})}{(C_{11} + C_{12})} = 130.12 \text{ GPa} \qquad (2)$$

The shear modulus for torsion about the <001> direction is given by [4]:

$$G = 1/((1 - \delta)\, S_{44}) = C_{44}(1 - \delta) = 79.17 \text{ GPa} \qquad (3)$$

for the accepted value of δ, the shear coupling factor, of 0.005.

The above values are very similar to those cited in [4] (E = 130.2 GPa and G = 79.4 GPa respectively, citing [7] and [8]).

These values are less than 0.5% different from the determined experimental values in Table 4. This value is similar to the level of uncertainty attributable to geometrical and dimensional measurement uncertainties on thin bar samples where the principal error is the beam thickness.

It will be noted that in Table 1, the computed apparent Poisson’s ratio is negative, and this is purely a result of using the isotropic calculation $\nu = (E/2G) - 1$. Strictly, for the orientation employed for the present test-pieces, Poisson’s ratio for strains transverse to the <001> direction should be defined as $-S_{12}/S_{11}$ [9]. Using the literature data given above, the correct value can be calculated as 0.278. It is not possible to convert the experimental data into the same format because only a single orientation of test-piece was measured, and this does not afford the opportunity to obtain S (see Equation 1) experimentally and hence to compute S_{12}.

Table 4 – Elastic moduli determinations on the three samples

		Sample #1			**Sample #2**			**Sample #3**		
Strike mode	**ν, assumed**	***E*, GPa**	***G*, GPa**	**ν, exptl.**	***E*, GPa**	***G*, GPa**	**ν, exptl**	***E*, GPa**	***G*, GPa**	**ν, exptl.**
Flexural, centre strike	0.25	130.48			128.79			129.98		
		130.46			128.78			129.98		
		130.48			128.79			129.98		
		130.48			128.78			129.98		
		130.48			128.78			129.98		
	Mean	**130.48**			**128.78**			**129.98**		
	std dev	**0.01**			**0.01**			**0.00**		
Flexural, end strike	0.25	130.48			128.78			129.98		
		130.48			128.83			129.99		
		130.49			128.78			129.98		
		130.47			128.78			129.99		
		130.47			128.83			129.99		
	Mean	**130.48**			**128.80**			**129.99**		
	std dev	**0.01**			**0.03**			**0.01**		
Torsional, end strike	0.25	131.77	78.82	-0.164	127.44	77.86	-0.182	129.24	78.87	-0.181
		131.40	78.81	-0.166	128.54	77.86	-0.175	129.02	78.87	-0.182
		131.27	78.82	-0.167	128.24	77.97	-0.178	128.97	78.87	-0.182
		131.31	78.82	-0.167	128.47	77.85	-0.175	129.72	78.87	-0.178
		131.44	78.82	-0.166	128.65	77.87	-0.174	129.12	78.87	-0.181
	Mean	**131.44**	**78.82**	-0.166	**128.27**	**77.88**	-0.180	**129.21**	**78.87**	-0.181
	std dev	**0.20**	**0.00**	0.001	**0.49**	**0.05**	0.00	**0.30**	**0.00**	0.002
Torsional, centre strike	0.25				128.82	78.63	-0.181	130.03	78.77	-0.175
					128.82	78.65	-0.181	130.03	78.81	-0.175
					128.84	78.65	-0.181	130.03	78.80	-0.175
					127.58	78.65	-0.189	130.01	78.75	-0.174
					128.84	78.65	-0.181	130.03	78.80	-0.175
	Mean				**128.58**	**78.65**	-0.182	**130.03**	**78.79**	-0.175
	std dev				**0.56**	**0.01**	0.003	**0.01**	**0.03**	0.000
Longitudinal strike	0.25	129.92			129.43			129.61		
		129.91			129.42			129.60		
		129.90			129.42			129.61		
		129.91			129.43			129.60		
		129.91			129.43			129.60		
	Mean	**129.91**			**129.43**			**129.60**		
	std dev	**0.01**			**0.01**			**0.01**		

5 CONCLUSIONS

The elastic moduli and effective Poisson’s ratio of three single-crystal silicon ‘reference’ samples have been determined using the impact excitation method.

Although the traceability of the literature data for the tensor stiffnesses of single-crystal silicon is unclear, its pedigree suggests that they are the accepted data for this material, and this implies that the IMCE equipment employed in this work is capable of giving results for the flexural and torsional modulus to within 0.5% of the literature values.

Since the greatest error in making measurements with this equipment is in the geometry and surface finish of the test samples and in their dimensional measurement, it can be concluded that although there is an added degree of complexity in using a single crystal rather than an isotropic material, the use of such a reference material provides an adequate level of traceability on the proper behaviour of the software system within the commercial impact excitation system.

ACKNOWLEDGEMENTS

Dr Phil Williams of Mettler –Toledo (UK) and Mr Urs Joerimann of Mettler-Toledo (Switzerland) are thanked for their supply of the test material.

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[3] Kim, J., Cho, D., Muller, R.S., “Why is (111) silicon a better mechanical material for MEMS”, *Proc. 11th Int. Conf. Solid State Sensors and Actuators”, Münich, Germany, 10-14 June 2001*, Digest of technical papers, edited by Obermeier, E, Springer-Verlag, Berlin, pp 662-5.

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Annex A – Single crystal silicon reference material certificates

Certificate

ME Number: **ME-51141618**

Item	Single Crystal Silicon Reference Sample
Supplier	PB-Technik AG CH-8702 Zollikon, Switzerland
Material	Silicon, strips orientation <100>
Theoretical modulus [1]	130.2 [GPa]

Dimensions:

Length / Tolerance	100 [mm]	± 1 [mm]	
Width / Tolerance	10 [mm]	± 0.2 [mm]	
Height / Tolerance / Parallelism	1 [mm]	± 0.03 [mm]	± 0.01 [mm]

Method:	**3-point bending**
Clamping length	90 [mm]
Frequency	1 [Hz]
Temperature	20 °C
Force amplitude	100 [N]
Displacement amplitude	20 [μm]
Offset type	Constant current mode
Offset value	3 N

Expected values :

Sample temperature:	10 - 30 [°C]		
Modulus: min. / typical / max.	120 [GPa]	128 [GPa]	135 [GPa]

Handle with care. The Si reference sample is extremely brittle and should therefore only be taken out of the container for calibration purposes.

The sample dimensions (height and width) must be accurately determined before the measurement.

Enclosure: Certificate of Manufacturer
Certificate of Supplier

[1] J. Kim, D. Cho, R. S. Muller, "Why is (111) silicon a better mechanical material for MEMS" in Proceeding of The 11th International Conference on Solid-State Sensors and Actuators, Munich, Germany, June 10 – 14, 2001

OTK-4

TEST CERTIFICATE

Customer name -	
Commodity description 150.0+/-1 (mm) silicon ingot	Customer spec N : 6400
Certification N:5516	Certification date : 2003 June, 14
Ingot lot N: 05200326/0-A	Date of manufacture : 2003 June, 09
Ingot conductivity: P ASTM F42	Dopant : Boron

TEST RESULTS	ASTM	CUSTOMER SPEC	ACTUAL
Diametr (mm)			150.9-150.9
Resistivity(ohm/cm)	F43		0.70-0.60
RRV%	F81		
Primory flat (mm)	SEMI		
Dislocation (cm^-2)	F1725		0
Boron (X10^14at/cm^3)			
Ingot length (mm)			105
Massa (kg)			4.36
Orientation	F26	(100)	(100) 0 12' 0 08'

Marushan	gauge	2003 June, 14
NAME	TITLE	DATE

TEST CERTIFICATE

Customer name -			
Commodity description 150.0+/-1 (mm) silicon ingot		Customer spec # 6400	
Certification # 5516		Certification date: 2003 June, 14	
Ingot lot # 05200326/0-A		Date of manufacture: 2003 June, 09	
Ingot conductivity: N	ASTM F42	Dopant: Boron	
Test results	ASTM	Customer spec	Actual
Strips orientation			≤ 1.5°
Output control			