

# Synthetic moissanite from Russia

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**ABSTRACT:** Five faceted yellow, green, bluish-green and bluish-brown synthetic moissanites of Russian production are described. Polytypism is characterized by Raman spectroscopy. Gemmological properties as well as chemical and spectroscopic properties in the UV-visible range are given. The samples were found to be of 6H- as well as 4H-SiC single crystals, and the variable coloration is caused by various amounts of nitrogen in the lattice of the synthetic moissanites. The criteria already described for the distinction of colourless American synthetic moissanites from diamonds in general also apply for a microscope characterization of Russian samples, especially to distinguish them from coloured diamonds.

**Keywords:** colour origin, diamond imitation, polytype, Russia, spectroscopy, synthetic moissanite

## Introduction

Silicon carbide – moissanite – has been used since the last decades of the 19th century for technical purposes (Knippenberg, 1963). The growth of large crystals of synthetic moissanite by a sublimation technique from the vapour phase was first described by Lely (1955), but the crystals grown by this so-called Lely technique consist of a mixture of simultaneously grown hexagonal (H), rhombohedral (R) and cubic (C) polytypes (Knippenberg, 1963; Kawamura, 1965; Verma and Krishna, 1966). This disadvantage for the technical usage of the crystals is overcome by the use of oriented seeds of selected polytype (Tairov and Tsvetkov, 1978; 1981). By seeded growth of silicon carbide by sublimation from the vapour phase, it is possible to grow large single crystals consisting of only one silicon carbide polytype, e.g. 6H-SiC, 4H-SiC. This method is described in the literature as the

modified Lely technique (see, e.g., Yoo *et al.*, 1991; Tairov, 1996; Tsvetkov *et al.*, 1996; Maltsev *et al.*, 1996; Ohsato *et al.*, 1999; Müller *et al.*, 2000; Yakimova *et al.*, 2000).

The modified Lely technique is also applied for the production of colourless synthetic moissanite crystals used as diamond substitutes (Davis *et al.*, 1989, 1995; Nassau *et al.*, 1997; Nassau, 1999). The colourless synthetic moissanites grown in the United States for jewellery purposes by Cree Inc., Durham, North Carolina and distributed by Charles & Colvard (formerly C3 Inc.), Morrisville, North Carolina, consist of single crystals of one of the known hexagonal silicon carbide polytypes, namely 6H-SiC (Nassau *et al.*, 1997; Nassau, 1999), but the growth of other polytypes and/or differently coloured moissanites for gem purposes is also possible (Hunter and Verbiest, 1998a; 1998b; Schmetzer, 2000). By the addition of pairs of charge-compensating trace elements such as

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nitrogen and aluminium, completely colourless crystals can be produced (Carter *et al.*, 1998). Most recently, diamond-coated moissanite was described (Nassau *et al.*, 1999; Hammer and Schmetzer, 2000) and aluminium nitride is also mentioned as a possible new diamond substitute (Hunter, 2000).

Due to its gemmological properties, synthetic moissanite, 6H-SiC, is an ideal diamond imitation, with refractive indices of 2.648 and 2.691 and a hardness of 9  $\frac{1}{4}$  on the Mohs scale (Nassau *et al.*, 1997). Although strongly anisotropic, the thermal conductivities of synthetic moissanites are so close to those of diamonds that the commonly applied thermal probes react to synthetic moissanites as if they were diamonds (Chalain and Krzemnicki, 1999).

Since the introduction of moissanite as a diamond substitute in 1997, the material has been distributed widely and has appeared worldwide in gemmological laboratories as a diamond imitation, in some instances, even set in antique jewellery!

Synthetic moissanites for gem purposes are also produced in small quantities in

St. Petersburg, Russia (Balitsky, 2000a; 2000b), and already the first samples of this material have been seen in the gem trade (Longère, 2000; McClure and Moses, 2000). Recently, faceted synthetic moissanites of Russian production weighing more than 100 ct have been submitted to gemmological laboratories as natural diamonds (Lasnier, 2001). By courtesy of Mrs B. Schaeffer, a gemmologist from Detmold, Germany, the authors were able to study five samples of the Russian production. Gemmological, microscopic, chemical and spectroscopic properties of these five faceted gemstones are presented in this paper.

## Materials and methods

The research material consisted of five brilliant-cut synthetic moissanites, which range in colour from yellow, green (two), bluish-green to bluish-brown (Table I, Figure 1). The stones weigh between 0.381 and 0.601 ct. According to the supplier all samples were grown in St. Petersburg using, at least basically, the method described by Tairov and Tsvetkov (1978, 1981).

**Table I:** Gemmological properties of Russian synthetic moissanite.

Sample No.	Weight (ct)	Polytype	Colour	SG	Relative reflectivity*	Orientation of tube inclusions with respect to the table facet	Pleochroism**
1	0.442	6H	yellow	3.250	118	about 45°	extremely weak
2	0.562	6H	green	3.211	122	parallel	weak: yellowish green/green
3	0.601	6H	green	3.231	121	parallel	weak: yellowish green/green
4	0.381	6H	bluish-green	3.201	120	no tubes observed	moderate: yellowish green/blue-green
5	0.542	4H	bluish-brown	3.207	120	about 5°	distinct: greenish blue/brown

\* based on 100 for diamond

\*\* as seen with a dichroscope



For the determination of moissanite polytypes Raman spectra were recorded in the 100-1800  $\text{cm}^{-1}$  range using a Renishaw Raman System 1000 spectrometer equipped with a CCD Peltier detector and an argon ion laser (514.5 nm) with a power of 25 mW.

We measured the relative reflectivity of the samples with a Presidio Duotester, where the value 100 was calibrated as the reflectivity of diamond. Specific gravity was determined hydrostatically. Internal features of all samples were examined using a standard gemmological microscope in conjunction with brightfield, darkfield, and oblique fibre-optic illumination. Photomicrographs were taken with a Wild M8/MPS55 stereozoom microscope. Different types of inclusions were examined by micro Raman spectroscopy using the facility at the SSEF Swiss Gemmological Institute.

Qualitative chemical analyses were performed by energy dispersive X-ray fluorescence (EDXRF) using a Tracor Spectrace 5000 instrument. For the examination of colour and colour causes we recorded UV-visible range spectra of all samples using a Varian Cary 500 spectrophotometer in the 300-800 nm range.

### Determination of moissanite polytypes with Raman spectroscopy

Various techniques, e.g. X-ray crystallography, can be applied for the determination of silicon carbide polytypes. The examination of the Raman spectrum is one of the non-destructive methods which can be performed on cut gemstones without special preparation of the samples. Using a micro Raman spectroscopy facility, which is now available for routine investigations in some of the major gemmological laboratories, it is also possible to examine the samples with respect to homogeneity in different areas.

We performed Raman spectra on different facets and at different points on the larger tables of all samples including different orientations of the

faceted gemstones to the incident beam. Although the spectra obtained, especially the intensities of the main peaks in the 760 to 800 and the 960 to 970  $\text{cm}^{-1}$  area, were found to be strongly variable and related to the orientation of

the samples (Figures 2 and 3), we could not determine any inhomogeneity within the five samples. In other words, the samples were found to be single crystals without any admixtures of different polytypes.

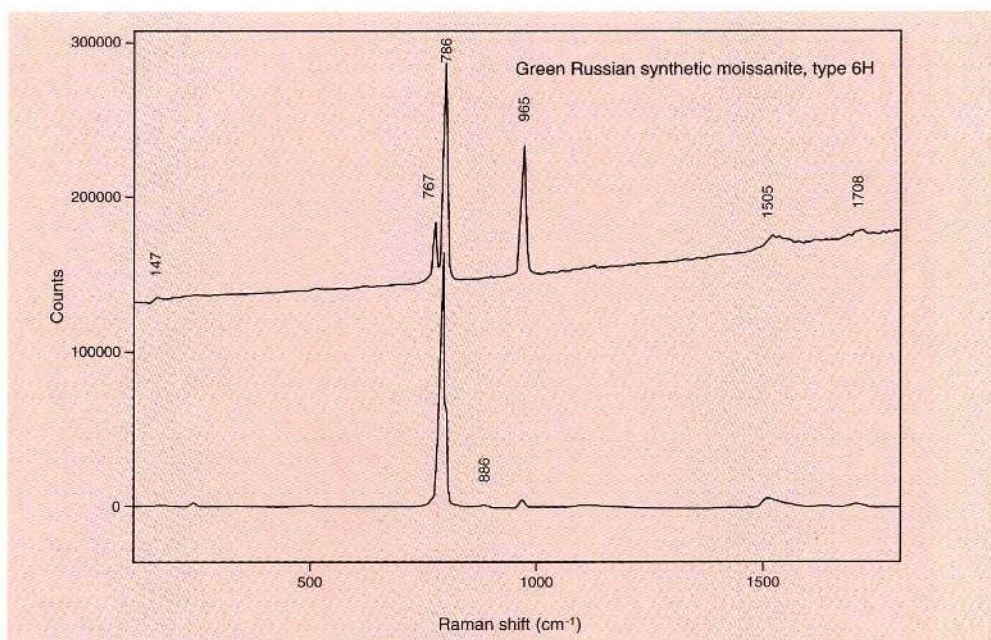
In all samples, the strongest Raman peaks above 700  $\text{cm}^{-1}$  were found at similar wave numbers. The spectra of four samples consisted of strong lines at 767, 786 and 965  $\text{cm}^{-1}$ , sometimes with an additional line at 795  $\text{cm}^{-1}$  (not shown in Figure 2). Weak lines above 1000  $\text{cm}^{-1}$  were also present. The spectra of the bluish-brown moissanite consisted of three strong lines at 778, 794 and 969  $\text{cm}^{-1}$ , again with two weak lines above 1000  $\text{cm}^{-1}$  (Figure 3). The Raman lines with weaker intensities below 700  $\text{cm}^{-1}$ , again were found to be different for the sample with bluish-brown colour (Figure 4). The main Raman lines for the first four samples in this range were determined at about 147, 238, and 504  $\text{cm}^{-1}$ , with one weaker line observed sometimes at 263  $\text{cm}^{-1}$ . The remaining sample, on the other hand, revealed lines with maxima at about 201, 263, and 609  $\text{cm}^{-1}$ , with an additional peak of weak intensity at 636  $\text{cm}^{-1}$ .

These data are consistent with numerous references describing Raman data of specific silicon carbide polytypes (e.g. Feldman *et al.*, 1968a; 1968b; Colwell and Klein, 1972; Harima *et al.*, 1995; Harima and Nakashima, 1996; Nakashima and Harima, 1997; Burton *et al.*, 1998; 1999) and indicate that four of our five samples belong to the 6H-SiC polytype, whereas the remaining faceted moissanite

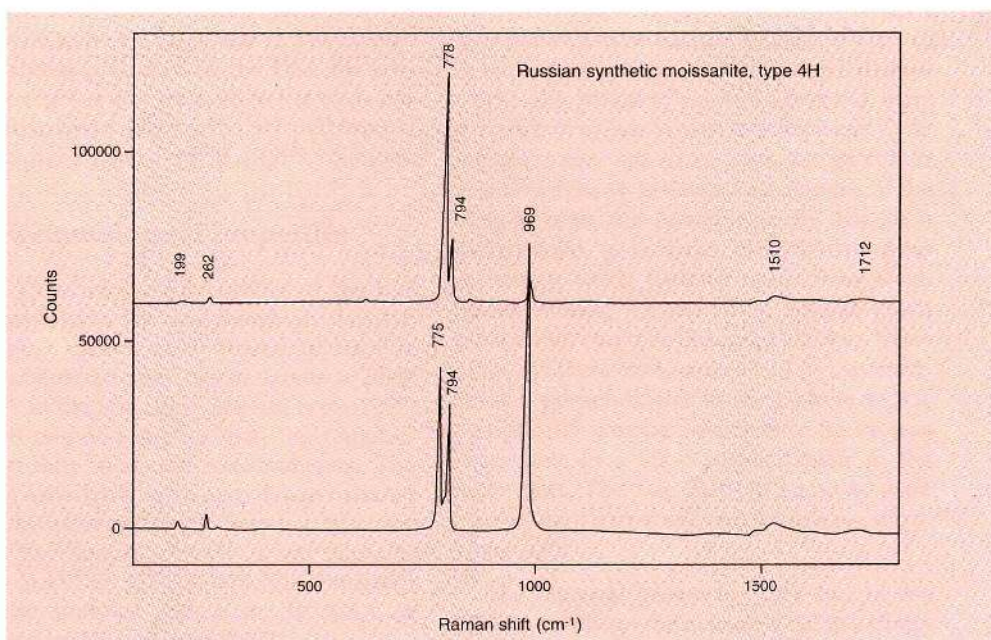


**Figure 1:** Faceted synthetic moissanites grown in St. Petersburg, Russia, by sublimation from the vapour phase. The samples range from 0.38 to 0.60 ct in weight and measure from 4.60 to 5.70 mm in diameter. Photo: © L.Kiefert, SSEF.



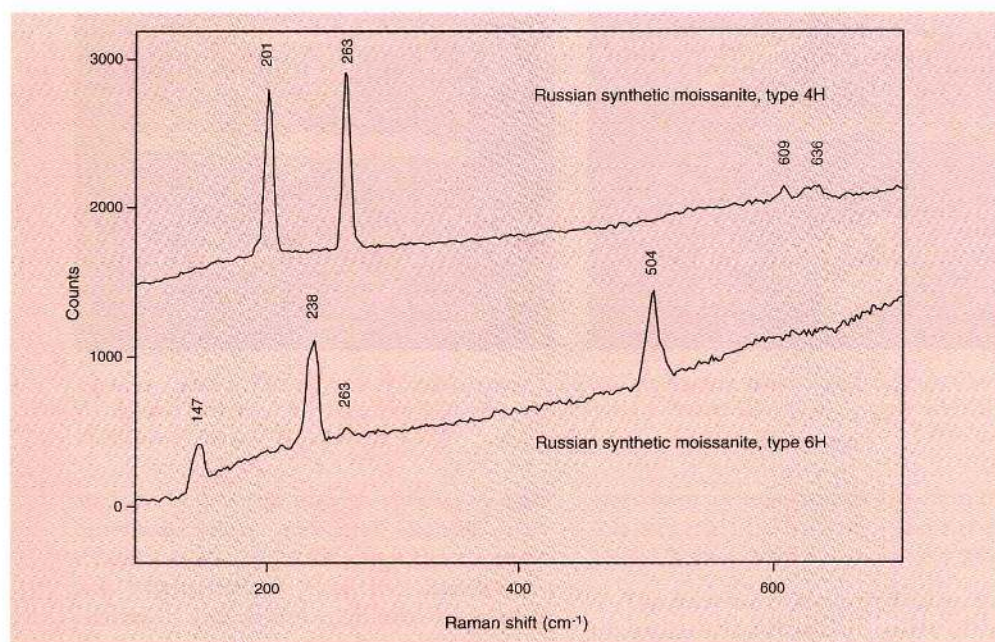


**Figure 2:** Raman spectra of synthetic moissanite (6H-SiC) in different orientations of the crystal with respect to the incident beam reveal a strong intensity variation of Raman lines.



**Figure 3:** Raman spectra of synthetic moissanite (4H-SiC) in different orientations of the crystal with respect to the incident beam reveal a strong intensity variation of Raman lines.





**Figure 4:** Raman spectra of different synthetic moissanite polytypes; the lines below 700  $\text{cm}^{-1}$  are useful for a distinction of 6H-SiC and 4H-SiC silicon carbide polytypes.

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was found to be 4H-SiC (Table I). The colourless material produced in the USA for jewellery purposes and released to the trade has been described so far as the 6H polytype of moissanite (Nassau *et al.*, 1997; Nassau, 1999).

### Gemmological properties

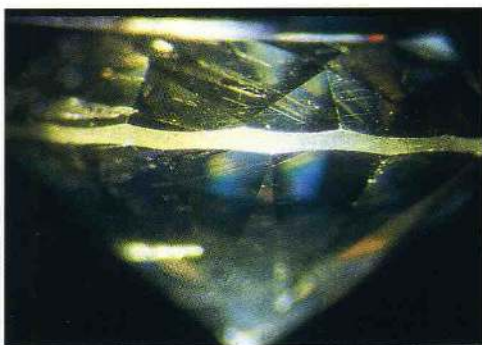
The gemmological properties of the five examined samples are listed in Table I. Refractive indices of synthetic moissanite were not determined. They are given as 2.648 to 2.691 in the literature (Nassau *et al.*, 1997) and are therefore higher than the range of commercially available refractometers. The relative reflectivity with an average value of 120 is distinctively higher than that of a diamond. The specific gravity of the samples ranges from 3.20 to 3.25, which is consistent with the samples grown in the USA as described by Nassau *et al.* (1997).

With the naked eye, only one of the samples which was determined as 4H-SiC

revealed different colours (blue and brown) in various directions of view. Using a dichroscope, the different colours observed were greenish-blue and brown (Figures 5 and 6). In the other four stones no variation of colour was observed in different orientations of the sample with the naked eye. By use of a dichroscope, a variable pleochroism from extremely weak (yellow sample) to weak (green stones) or moderate (bluish-green moissanite) could be observed (Table I). None of the synthetic moissanites had its *c*-axis orientated perpendicular to the table facet, i.e. no optically uniaxial interference figure was observable in a view perpendicular to the table facet. The five Russian samples were inert under short-wave and long-wave ultra-violet light.

The material grown by Cree Inc. in the USA currently on the market is cut in a direction with the table facet more or less perpendicular to the *c*-axis. This material has a colour range from near colourless, through



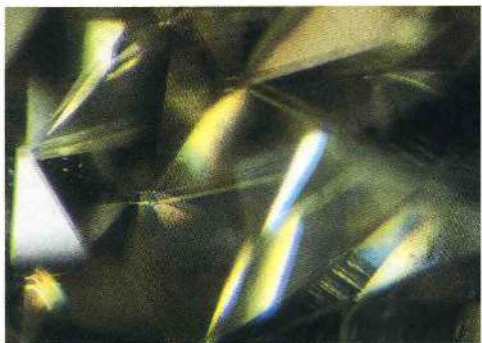


**Figures 5 and 6:** Pleochroism of bluish-brown synthetic moissanite which was determined as 4H-SiC by Raman spectroscopy. Figure 5 blue direction, Figure 6 brown direction. Sample is 5.40 mm in diameter. Photos: © H.A Hänni, SSEF.

slightly yellow or greyish-yellow to light brownish-yellow, grey and slightly green (Nassau *et al.*, 1997), although the commercial production of Cree Inc. focused on colourless and nearly colourless material in the first years. Near-colourless and grey samples were not encountered in the material of the present Russian production available to the authors. Pale green, bluish-green and grey colours have recently been encountered in faceted stones from the commercial American production submitted to the market (Sluis, 2001).

### Microscopic characteristics

The Russian stones show the typical doubling of facet edges (Figure 7) as described



**Figure 7:** Doubling of facet junctions in Russian synthetic moissanites. This is one of the most concise diagnostic features which is easily determined using a hand-lens or the microscope. Magnified 40 x. Photo: © H.A Hänni, SSEF.

elsewhere for moissanites grown in the USA (Nassau *et al.*, 1997; Chalain and Krzemnicki, 1999; Chalain, 2000; Shigley *et al.*, 2000).

The presence of elongated tubes, so-called micropipes, is a typical property of sublimation-grown 6H-SiC and 4H-SiC synthetic moissanites using a modified Lely technique (Takanaka *et al.*, 1996; Ohsato *et al.*, 1999; Müller *et al.*, 2000). One stone of our five synthetic moissanites is virtually inclusion-free (sample 4). Four of the five analysed stones contain elongated thin tubes or irregular cavities (Figures 8 and 9). In comparison to the Cree stones encountered in the SSEF Swiss Gemmological Institute as diamond imitations and described in the literature, most tubes appear thicker and are orientated parallel or slightly inclined to the table facet (samples 2, 3 and 5) as well as inclined at an angle of 45° to the table (sample 1). The Cree stones generally have thinner tubes that are orientated more or less perpendicular to the table, i.e. parallel to the *c*-axis. This orientation of tubes in American stones is responsible for an image which gives the impression that they are radiating around the centre of the stone (Chalain, 2000). This difference in appearance in stones from the two producers is caused by the different orientation of the optic axis within the faceted stones.

Small crystals or bubbles are common in four of the five stones (Figures 8 and 9). One stone (sample 2) shows growth lines that are orientated perpendicular to the tubes





**Figure 8:** Elongate tubes or irregular cavities in Russian synthetic moissanites; irregular bubbles and/or small crystals are also present; growth planes perpendicular to the tubes are also seen. Magnified 40 x. Photo: © H.A. Hänni, SSEF.



**Figure 9:** Elongate tubes or irregular cavities in Russian synthetic moissanites; irregular bubbles and/or small crystals are also present. Magnified 40 x. Photo: © H.A. Hänni, SSEF.

(Figure 8), and one of the synthetic moissanites (sample 1) contains a highly reflective hexagonal crystal or negative crystal with a hollow tube extending from it (Figure 10). Similar defects were described by Takanaka *et al.* (1996) in synthetic 6H-SiC.

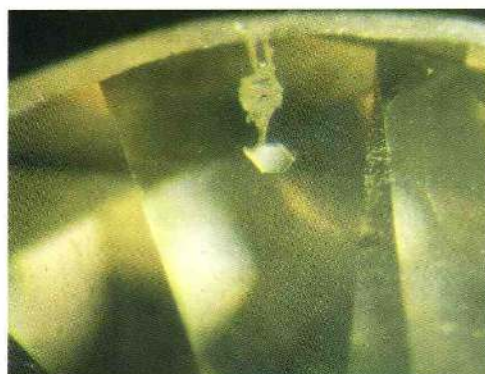
Analysis of elongated tubes, small crystals or bubbles, as well as the hexagonal crystal mentioned above, with micro Raman spectroscopy revealed only the common spectra of the host moissanite polytypes. Consequently, all encountered inclusions are likely to be either cavities or inclusions of moissanite crystals, possible in an orientation different from that of the host. However, it is also possible that these inclusions are phases that give only weak or no Raman signals. Although thick tubes, hexagonal crystals, or growth structures are not commonly observed in the synthetic moissanites from Cree, all these features were observed by Groenenboom (2000) in samples grown in the USA. They are, therefore, neither exclusively found in, nor characteristic of synthetic moissanite of Russian production.

### Chemical properties

According to Lely & Kröger (1958), hexagonal silicon carbide crystals owe their colour to impurities of N or P (green), or Al or B (blue). Nitrogen and aluminium are the commonly used dopants for the growth of silicon

carbide gem materials in different colours (Hunter and Verbiest, 1998a; 1998b).

The X-ray fluorescence spectrometer used can detect elements with atomic numbers above 11 (Na). Semi-quantitative chemical analysis of the five stones under investigation revealed, besides Si, no other element. Hence, no P or Al was detectable in our samples. This implies that these elements, if present, can only exist in traces below the detection limit of the instrument. The presence of B and especially N, on the other hand, cannot be discerned with the X-ray fluorescence facility applied.



**Figure 10:** Tiny moissanite crystal or negative crystal with an elongate tube extending from this inclusion in yellow synthetic moissanite from Russia. Magnified 30 x. Photo: © H.A. Hänni, SSEF.



## Spectroscopic properties in the visible and UV range

As a consequence of intense internal reflections due to the high refractive indices of moissanites, and of oblique orientations of the optic axis of the faceted samples, we were unable to orient the Russian moissanites properly to the incident beam of the spectrophotometer to obtain conventional polarized spectra parallel and perpendicular to the *c*-axis.

Absorption spectroscopy for yellow and green synthetic moissanites from Russia (samples 1, 2 and 3) revealed a slightly increasing absorption starting at about 600 nm towards the violet range and an absorption edge at about 410 nm (Figure 11). The two green samples showed an additional weak absorption band with a maximum at about 635 nm. In the bluish-green sample, both the absorption in the blue to violet range and the absorption band at 635 nm are distinctly increased in intensity, and a weak shoulder at 435 nm is also present (see again Figure 11).

Comparing these spectroscopic properties with published data of 6H-SiC (Lely and Kröger, 1958; Violina *et al.*, 1964; Biedermann, 1965; Ellis and Moss, 1965; Sugiyama *et al.*, 1996; Stiasny and Helbig, 1997; Lambrecht *et al.*, 1998; Limpijumnong *et al.*, 1999), all absorption features are consistent with those of nitrogen-doped synthetic moissanite. Nitrogen is the most common trace element in synthetic moissanites, originating at least partly from atmospheric air. In order to achieve colourless synthetic moissanites, great efforts are undertaken to grow almost nitrogen-free samples or to reduce the influence of nitrogen by charge compensating trace elements such as aluminium.

Spectra similar to those of our Russian samples 1 to 4 have already been published by Lely and Kröger (1958) for synthetic moissanites grown under different nitrogen-bearing argon atmospheres. The spectrum of our yellow sample 1 is consistent with that of

a sample grown at 0.01% N<sub>2</sub>, the spectra of the green samples 2 and 3 are consistent with the published spectrum of a sample grown at 0.1% N<sub>2</sub>, and the spectrum of the bluish-green sample 4 is similar to that of a synthetic moissanite grown at 10% N<sub>2</sub>. Consequently the colour of yellow, green and bluish-green synthetic moissanites is probably due to various concentrations of nitrogen in these 6H-SiC crystals.

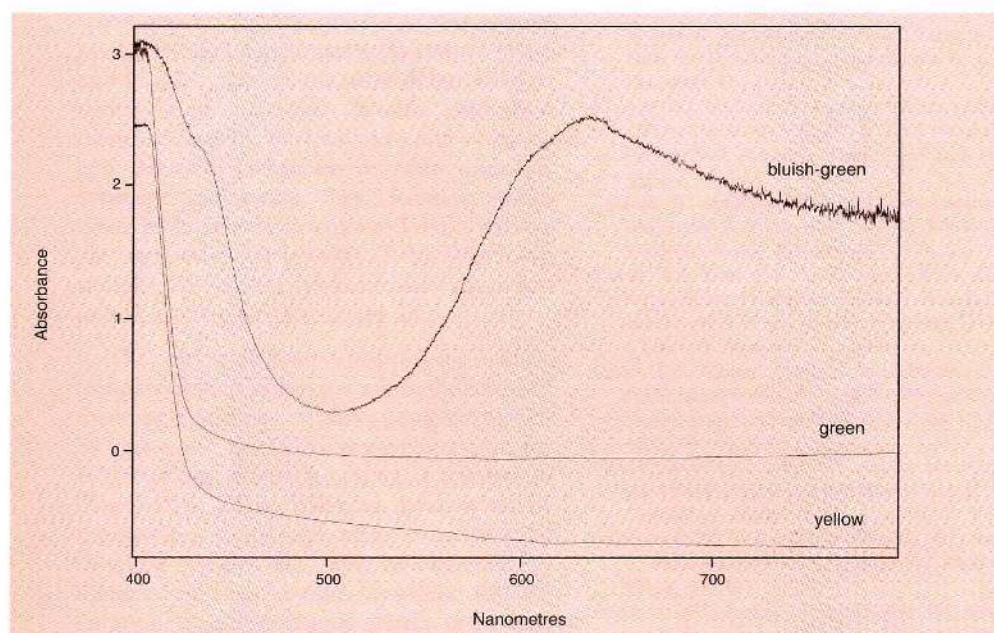
For the evaluation of the strong blue and brown pleochroism of sample 5, non-polarized spectra were recorded in those directions which showed these different colours (Figure 12). Both spectra revealed an absorption edge in the UV at about 370 nm. The spectrum in the blue-green direction revealed two strong absorption maxima at 566 nm (with a shoulder at 649 nm) and at 465 nm, and the spectrum taken in the brown direction consisted of two distinct absorption maxima at 553 nm and at 458 nm.

A shift of the absorption edge from the violet range in 6H-SiC to the ultraviolet in 4H-SiC is consistent with literature data (Choyke and Patrick, 1961; de Oliveira *et al.*, 1996). The spectra of the Russian sample 5 taken in different orientations are also consistent with reference data for nitrogen-doped synthetic 4H-SiC (Biedermann, 1965; Lambrecht *et al.*, 1998; Limpijumnong *et al.*, 1999). Comparing these literature references and the polarized absorption spectra pictured in these papers with our spectra it may be interpreted that the spectrum taken in the blue direction consists of superimposed polarized spectra with components parallel and perpendicular to *c*, and the spectrum taken in the brown direction represents a polarized spectrum perpendicular to *c*. Thus, the colour and pleochroism of blue to brown synthetic moissanite is probably caused by traces of nitrogen in 4H-SiC.

## Conclusions

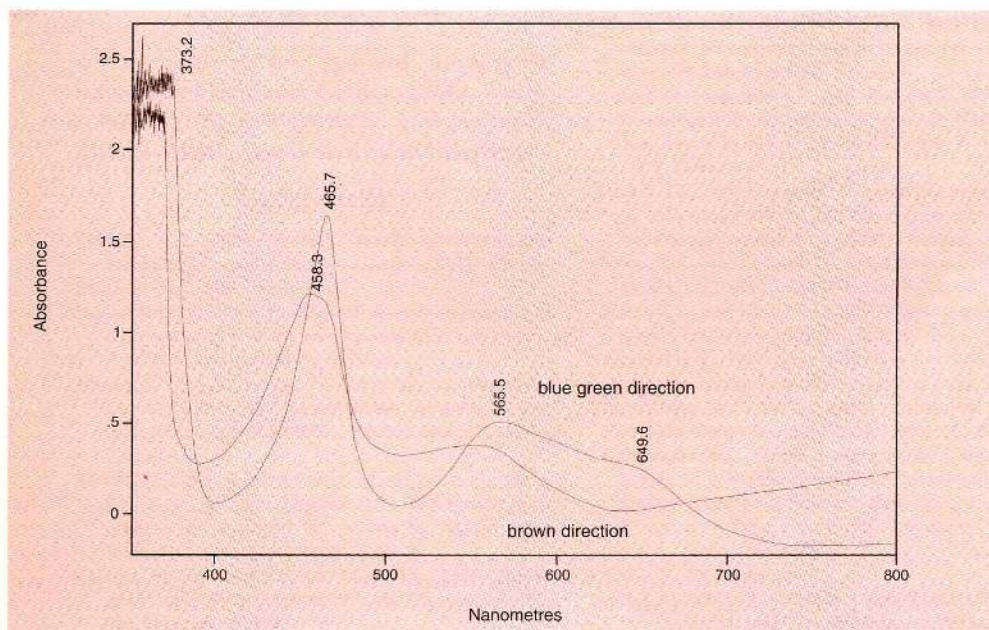
Hexagonal silicon carbide single crystals of 6H or 4H polytype have been grown in St. Petersburg, Russia, using the modified Lely





**Figure 11:** Absorption spectra of yellow [bottom], green [middle] and bluish-green [top] synthetic moissanites (6H-SiC) doped with different amounts of nitrogen.

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**Figure 12:** Absorption spectra of bluish-brown synthetic moissanite (4H-SiC) doped with traces of nitrogen recorded in the directions of the blue-green coloration and the brown coloration.



technique by sublimation from the vapour phase. Faceted gemstones with yellow, green and bluish-green colour were determined as synthetic 6H-SiC, the variable coloration being caused by various amounts of nitrogen. As with colourless or near-colourless synthetic moissanite, these Russian stones could be mistaken for coloured diamonds by the inexperienced jeweller. A bluish-brown sample with distinct pleochroism was determined as nitrogen-doped 4H-SiC.

The physical properties of moissanite samples of both polytypes are in the range of those of colourless or almost colourless 6H-SiC grown in the USA for jewellery purposes. Doubling of facet junctions is commonly observed in all samples. In most synthetic moissanites, elongated tubes or somewhat irregular cavities may also be observed by microscopic examination. However, the orientation of these characteristic inclusions tends to be sub-parallel to the table facet in contrast to a near perpendicular orientation in American synthetic moissanite material seen normally on the market.

### Acknowledgement

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