SEM Studies of Bleached and Polymer-impregnated Jadeite

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INTRODUCTION

Bleached jadeite or B-jade has provided many problems for the jewellery trade in the east Asian region[1,2]. Most B-jade comes from Hong Kong[2], where manufacturers have perfected techniques for enhancing the quality of all grades of jadeite.

Fristch et al.[1] have detailed basic techniques for identifying B-jade. Some recommended techniques, such as determination of relative density (specific gravity) and examination under long wave ultraviolet light, do not assist identification. To date, the most conclusive differentiation between natural coloured jadeite and bleached polymer- impregnated jadeite, can be made with infra-red spectroscopy[1]. Observation of absorption peaks around 3,000 cm^{-1} in the infra-red region of the electromagnetic spectrum, identify polymer-impregnated B-jade. Furthermore the presence of a polymer in a jadeite indirectly implies that the jadeite has been bleached before impregnation.

As enterprising manufacturers are always on the look-out for substitute fillers, bleached wax impregnated jadeite has recently been detected by infra-red spectroscopy[3]. In this paper, the authors explore the feasibility of using the scanning electron microscopy (SEM) to differentiate natural jadeite from bleached polymer-impregnated (treated) jadeite.

MATERIALS, METHODS AND RESULTS

Gemmological Investigations

Twelve green jadeites (Fig. 1), four natural coloured polished green cabochons, and eight bleached polymer-impregnated green polished jadeites, were studied using basic gemmological instruments. The results of this investigation are summarised in table 1. In addition, two rough jadeite pebbles, with weathered brownish 'skins' (Fig. 2), were examined to determine their ultraviolet fluorescence characteristics.

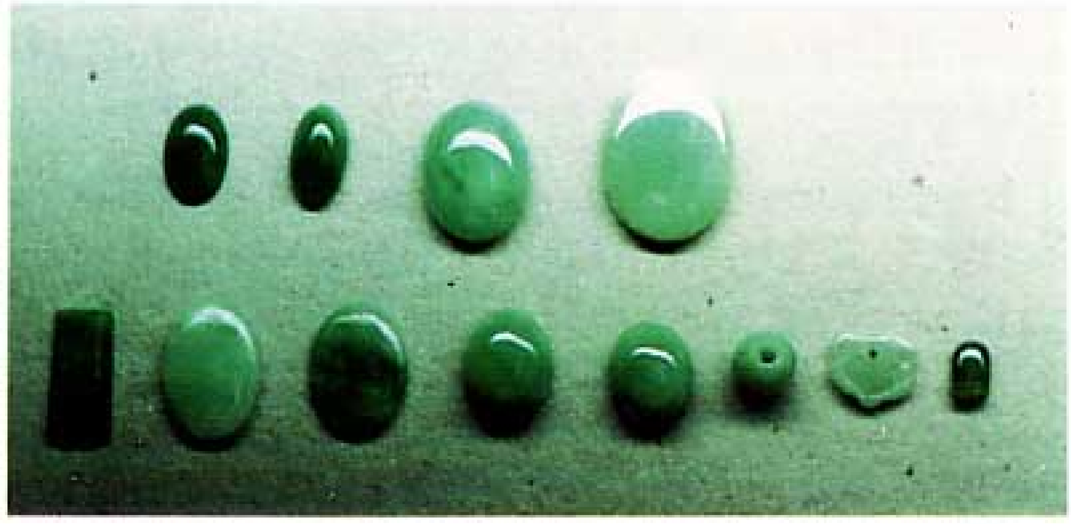

Fig. 1. Eight pieces of bleached polymer-impregnated jadeite (lower row), and four pieces of natural jadeite (upper row). For identification purposes the jadeites have been numbered left to right 1-4 (upper row), 5-12 (lower row).

Fig.2. Two weathered jadeite pebbles, each displaying a polished surface.

RESULTS

Refractive index

All 'spot' refractive indices were found to be within the mean jadeite range of 1.66.

Prism spectroscope

All jadeites exhibited the typical jadeite absorption spectra of a strong absorption band at 437 nm. In some light-medium to intense green jadeites graded 'venetian blind' absorptions at 630, 655, and 690 nm were observed. Natural coloured and bleached polymer-impregnated jadeites had identical absorption spectra.

Specific gravity

Bleached polymer-impregnated jadeites tended to float on a heavy liquid of 3.32 density. Only jadeites 8 and 9 sank slowly in this heavy liquid. All four natural jadeites sank slowly in the same heavy liquid.

Ultraviolet light

Under long wave ultraviolet light irradiation bleached polymer-impregnated jadeite tended to fluoresce a weak to moderate chalky blue. Three jadeites (5, 8, 9) were found to fluoresce a weak to moderate chalky yellow, while one jadeite (12) did not fluoresce. In contrast, all natural jadeites were inert to long wave ultraviolet light.

It is of interest to note that sawn polished cross sections of the two rough jadeite specimens (Fig. 2) fluoresced a rather strong chalky white along all weathered edges of the jadeite. More detailed observation revealed that this fluorescence was emitted by a whitish substance that was concentrated along fractures in or along the edge of the jadeite. This material is likely to consist of a decomposition product of jadeite - possibly kaolinite[8]. Therefore, the demonstrated ultraviolet light fluorescence of the skin on jadeite pebbles limits usefulness of jadeite's ultraviolet fluorescence as a determinant of its bleached polymer- impregnated status.

Binocular microscope

When examined with a gemmological microscope and pin-point incident illumination, the surface of bleached polymer-impregnated jadeite tended to display what has been described as either a 'cracked appearance'[1], or a 'spider-web effect' [2]. This surface texture was sharply contrasted by that displayed by natural jadeite. In natural jadeite observable surface cracking was minimal[1,2]. One B-jade (12) did not display a 'cracked appearance', but was found to float very rapidly in 3.32 liquid, thus implying that this jadeite was suspected of being treated. This piece of treated jadeite was later analysed by SEM and found to have a very fine interlocking granular crystal structure, but displayed the surface characteristics of a bleached polymer-impregnated jadeite.

SCANNING ELECTRON MICROSCOPE

The polished surfaces of selected specimens of naturally coloured jadeite and B-jade were examined by SEM under the following operating conditions: operating voltage - 100 ± 5v, accelerating voltage - 20Kv, gun filament current -

Sample	RI (spot)	SG (3.32)	UV/LW	Spectroscope (nm)	Other Observations
1 to 4	1.660	Sink	None	437 Green: 630, 655, 690	From light to medium green oval shape cabochon. Some green colour veins or patches.
5	1.660	Float	Moderately chalky yellow	437 Green: 630, 655, 690	Medium-light green with one end partially burned. Polymer treated jadeite tends to turn brown or light brown when heated by jewellers' torch (see the end of the table).
6	1.660	Float	Very weak chalky blue	437 Green: 630, 655, 690	Very light green with some green colour veins, surface cracked like effect is very prominent under reflected lighting.
7	1.660	Float	Very chalky blue at the girdle	437 Green: 630, 655, 690	Medium-light green oval flat cabochon, surface cracked like effect or spider web like pattern.
8	1.660	Sink	Weak light yellow	437 Green: 630, 655, 690	Light green oval high dome cabochon, shows surface cracked-like effect, 5 years in collection, some yellow and white spots appearing near the surface. The green colour remains the same.
9	1.660	Sink	Weak light yellow	437 Green: 630, 655, 690	Light green oval high dome cabochon, shows surface cracked-like effect, 5 years in collection, some yellow and white spots appearing near the surface. The green colour remains the same.
10	1.660	Float	Moderately chalky blue	437 Green: 630, 655, 690	Very light green round bead with a hole, showing cracked-like effect on the surface.
11	1.660	Float	Weak chalky blue	437 Green: 630, 655, 690	Very light green fancy carving pendant showing crack-like effect on the surface.
12	1,660	Float	None	437 Green: 630, 655, 690	Medium green, part of a broken piece of saddle shape jade, very smooth surface with no pit or cracked-liked effect, 4 years in collection with no observable change in colour.

Table 1. Gemmological data and other observations on natural and bleached polymer-impregnated jadeite.

52-55μA, filament - tungsten hairpin, electron detector - backscattered photomultiplier tube, vacuum mode - 10^{-2} Pa, resolution - 7-10 nm at 20 Kv, working distance - 20-30 mm, image mode - Compo-topo mix, magnification - x150 except for the fine textured jadeite.

RESULTS

Natural jadeite (Fig. 3) displayed a compact interlocking microgranular structure. Grain boundaries of natural jadeite were still closely approximated when examined at x2,000.

In bleached polymer-impregnated jadeites intense electrical charging occurred along intergranular fractures or grain boundaries between microcrystals. This increased charging occurred where polymer accumulated in acid bleached spaces (Fig. 4). In contrast, minimal charging occurred on the surface of those jadeites that had waxed external polished surfaces.

In acid bleach polymer impregnated jadeites the somewhat indistinct etched gaps between grain boundaries appeared rather distinct under SEM, the jadeite being classified as being either of the granular or fibrous type[4]. At x150 magnification, granular textured bleached polymer-impregnated jadeite had the appearance of dried-up mud flat (Fig. 5). In contrast, fibrous textured bleached polymer-impregnated jadeite had the appearance illustrated in figure 6.

Although binocular gemmological microscopes are commonly used to examine the external surface of polished jadeites, the SEM gives better contrast and resolution for identifying defects in the polished surfaces of jadeites. (Compare figures 5 & 6)

Fig.3. Compact interlocking grain boundaries of natural jadeite. (x2,000)

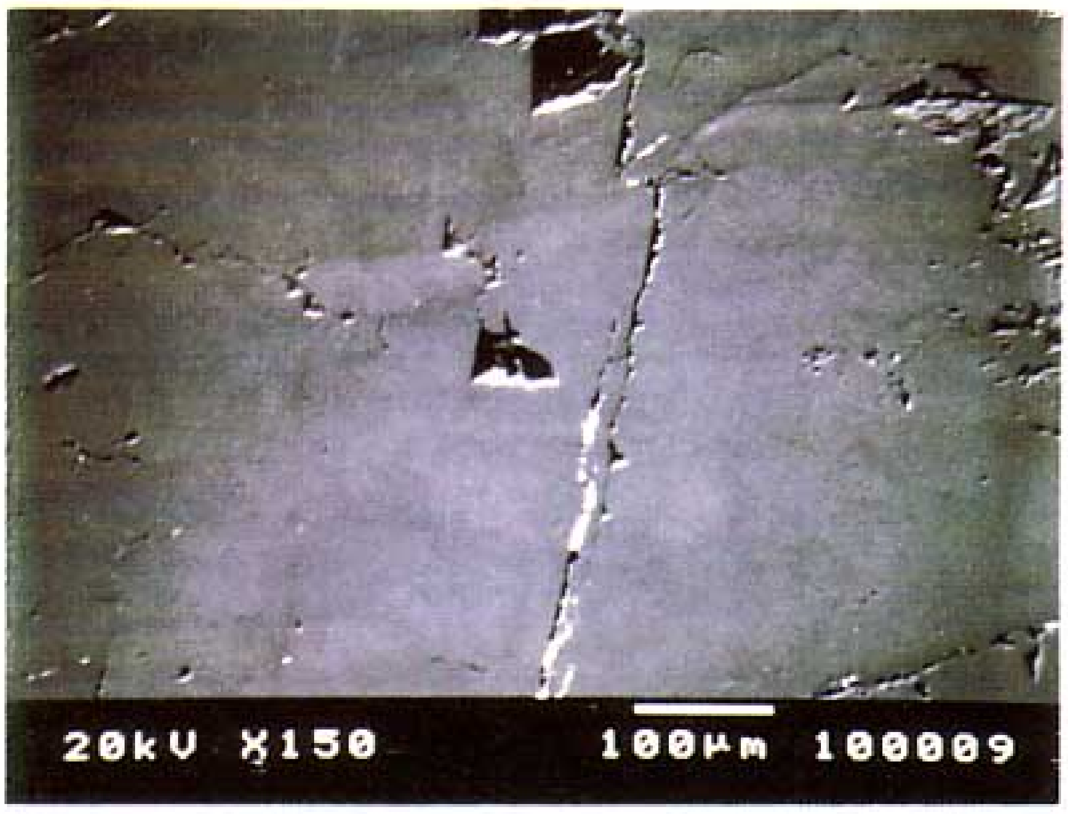

Fig.4. Electrical charging (bright white line) of polymer infilled grain boundaries and defects on the surface of a bleached polymer-impregnated jadeite. (x150)

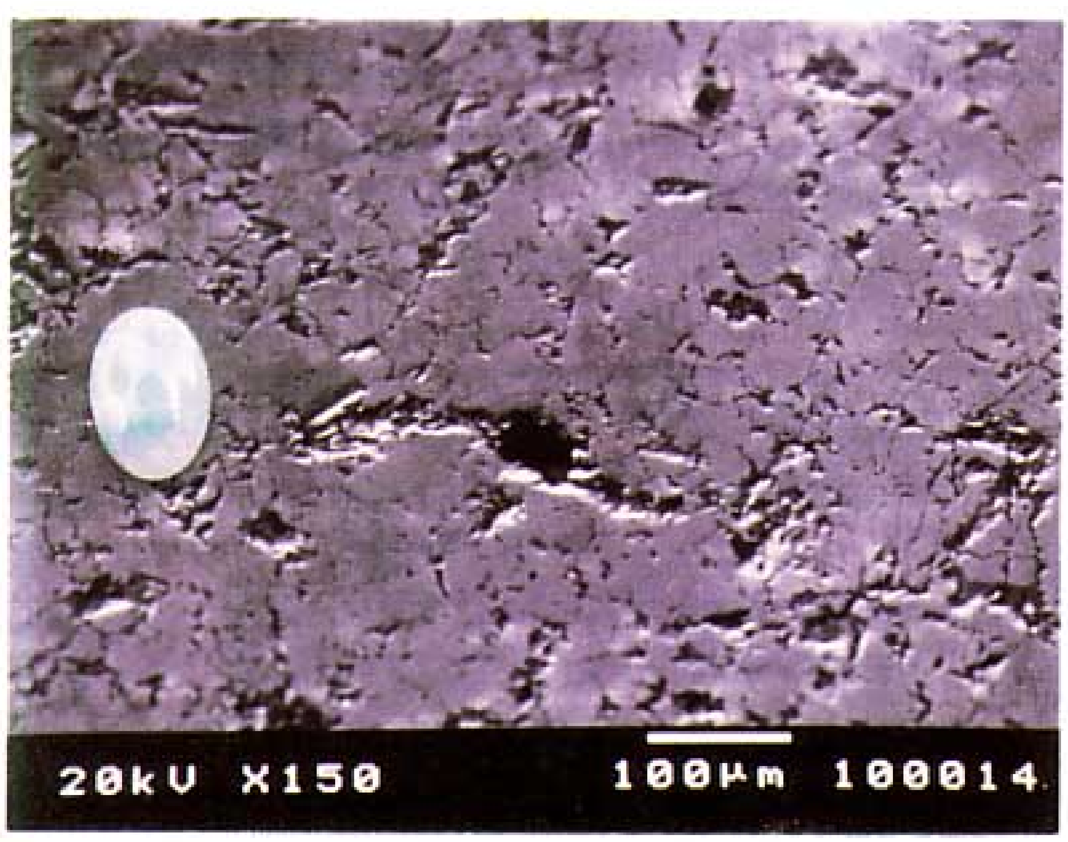

Fig.5. SEM appearances of B-jade displaying a 'dried out mud flat' texture. Note that the grain boundaries are more defined than when viewed with the light microscope. (x150)

Jadeite #12, a fine grain B-jade, had a very fine microgranular texture. Its treatment could not be detected with either the x10 hand lens or the gemmologist's binocular microscope. Even x150 magnifications with the SEM could not resolve the etched polymer-filled intergranular boundaries of this jadeite. However, SEM examination of the surface of this jadeite at x500 immediately revealed the widened intergranular boundaries of this etched wax-impregnated jadeite (Fig. 7).

When the sawn weathered jadeite pebbles (Fig. 2) were examined under the SEM, polished surfaces of the rough jadeite showed white patches of weak surface charging due to wax (Fig. 8). In addition, weak charging was visible along the boundaries of interlocking microcrystals. The weathered skin of the pebbles consisted of a rather indistinct mixed mass of material of undetermined composition.

CONCLUSION

For many years the SEM has been used by scientists to study the surface characteristics of materials such as rocks and minerals[5], biological materials[6], and integrated computer chips[7]. In this investigation the authors explored the feasibility of using the SEM to discriminate natural jadeite from acid bleached polymer-impregnated jadeite.

This investigation has revealed that jadeites, that had been chemically bleached and subsequently impregnated with polymer, displayed wider (etched) grain boundaries, fractures, and surface defects when examined with the SEM. In contrast, grain boundaries in natural jadeite were not prominent. The most identifying photomicrographs of microgranular boundaries in jadeite were obtained by simply tilting the specimens before examining their surfaces with the SEM at magnifications of from x150 to x500.

As the polymer impregnator, used to fill etched fractures and grain boundaries in polymer-impregnated jadeite is electrically non-conducting, the surface of the impregnator was intensely charged under the operating scan. This charging on scanning was recorded on the SEM photomicrograph as bright white lines surrounding grains or infilling fractures and surface defects on the jadeite, thus providing a clear indication that etched fractures, defects and/or microgranular boundaries on the surface of the jadeite had been impregnated with polymer.

The intense surface charging that identified polymer impregnation should not be confused with the rather mild surface charge generated, when a waxed jadeite surface is examined under the SEM. Wax has the SEM appearance of white 'clouds' or patches.

As intense surface charging was consistently observed on all jadeites that had been bleached and polymer- impregnated, the authors believe that SEM examination will prove to be useful to confirm other tests commonly used in the jewellery trade to discriminate treated from natural jadeite.

The authors concede that the SEM is an expensive microscope to purchase and operate. Additionally, access to SEMs is restricted for these microscopes are only likely to be available at Universities, scientific laboratories and a few gemmological laboratories across the world. As the SEM will achieve positive identification of a suspect bleached polymer-impregnated jadeite in under ten minutes, its use is recommended. Undoubtedly the sensitivity of this technique of discrimination could be enhanced by incorporating an electron gun (EXD detector) into the SEM. Then, trace elements, trapped in and around the interlocking micrograins of jadeite, could be chemically analysed and perhaps used to further discriminate a treated jadeite.

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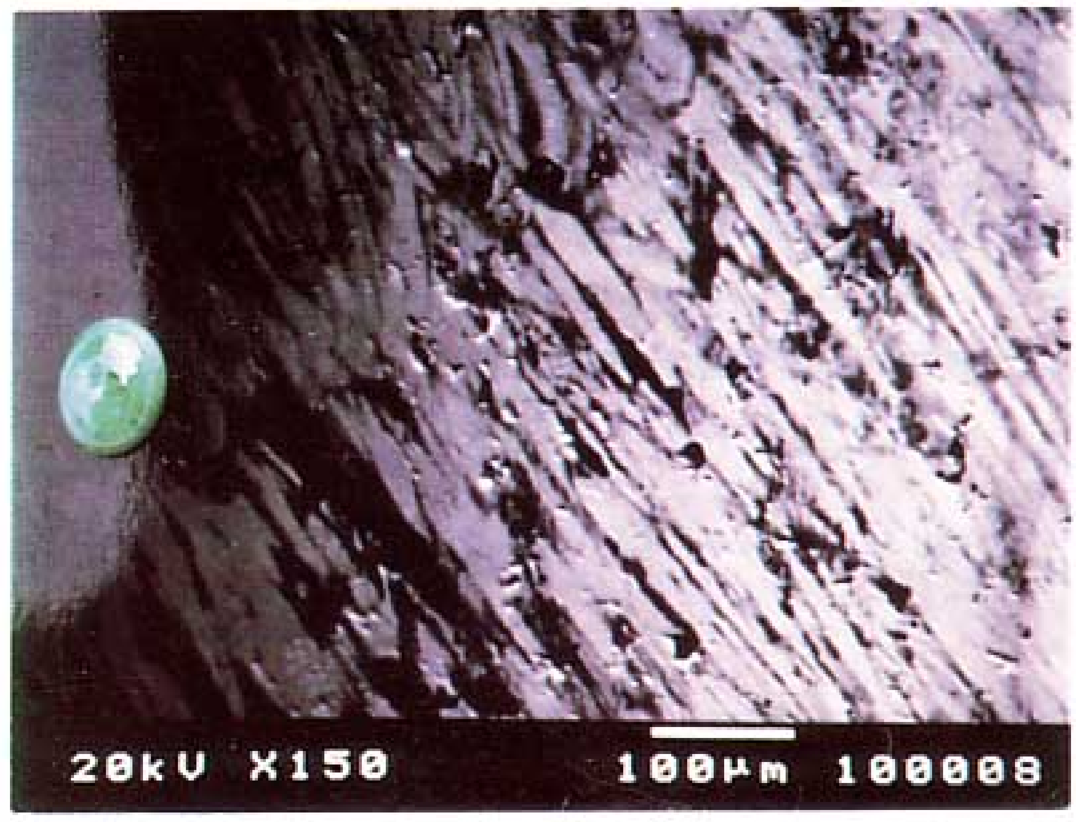

Fig.6. SEM appearance of B-jade displaying a loose fibrous texture. Note the wider gap between grains in this jadeite (Compare figure 5). (x150)

Fig.7. SEM appearance of B-jade #12 illustrating that x500 magnification was required to resolve micrograin boundaries of this jadeite. Notice the charging of surface wax represented by the white cloud-like patches in the right hand corner of the photomicrograph. (x500)

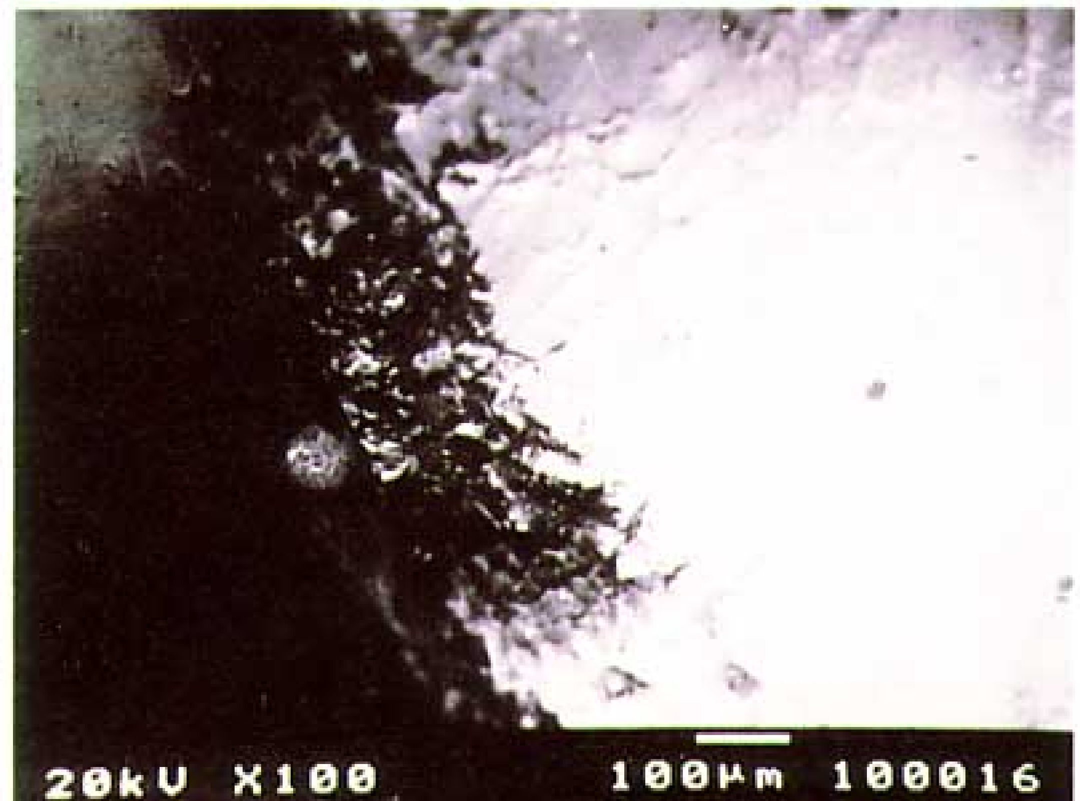

Fig.8. SEM appearance of the polished surface of one of the weathered jadeite pebbles. Note weak charging due to wax has the appearance of white patches. (x100)