Microstructural and topographical changes of Ni-P plated moulds in glass lens pressing

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Abstract: The changes in microstructure and topography of nickel-phosphorus (Ni-P) plated steel moulds in a glass lens moulding process were investigated by heating tests. The results show that, at a high temperature, Ni atoms diffuse from the inner Ni-P plating layer into the outer release agent coating consisting of precious metals. The atomic diffusion leads to partial surface swelling, which makes the mould surface rough and cloudy. The diffusion is influenced by the temperature, interface structure and stress state of the moulds. These findings provide references for developing new mould materials and for improving the service life of the moulds.

Keywords: glass moulding; mould structure; surface coating; Ni-P plating; atomic diffusion; interface phenomenon.

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1 Introduction

Optical and optoelectronic products, such as digital cameras, mobile phones, and high-density optical disc systems use many optical lenses. Generally, these optical lenses are made of plastic or glass. A characteristic of plastic lenses is that they are less expensive. Conversely, the features of glass lens are that they have a high refractive index, high permeability of light and good stability to environmental changes. Of late, glass lenses are increasingly necessary in the field of high-pixel digital cameras and in cameras used in mobile phones (Kinoshita, 2006). Another attention-attracting trend is the development of compound lenses, namely, lenses that combine the refractive and diffractive functions of light. Compound lens can consolidate the functions of two lenses into one, thus contributing to the miniaturisation and thinning of optical units. The substrate material used in compound lens is also changing from being plastic to glass. Compound lens made of glass contributes greatly to both the miniaturisation and the cost reduction of digital optical products (Tanaka et al., 2004; Sohn et al., 2006). The demand for optical lenses in the car manufacturing industry will also increase in the future as they are needed from the point of view of being stable to environmental changes

such as the temperature and humidity, etc. Therefore, reducing the cost of mass producing glass lens has become a necessity.

In general, industrial aspherical lenses made of glass are produced using press moulding technology. The high cost of glass lenses is caused by the low productivity of the glass moulding system and the high cost of the moulds. Refractive/diffractive compound lenses made of glass have not been put into practical use yet because of the difficulty in fabricating diffractive pattern moulds. Conventionally, moulds for glass lenses are made of cemented carbide or silicon carbide, which are high-hardness materials and must be ground and polished (Tanaka, 2004). The mould grinding, polishing, shape measurement and shape compensation process all need to be repeated in order to achieve the accuracy needed in the moulded end product. These complicated processing steps, therefore, raise the manufacturing cost of the moulds and then, in turn, the actual glass lenses. Reducing the cost of the mould processing is one of the most important issues in reducing the cost of glass lenses.

In a previous paper (Masuda et al., 2007), the present authors demonstrated that moulds made of steel plated with nickel-phosphorus (Ni-P) alloy can be used for moulding glass lenses. Ni-P plated moulds can be easily fabricated through ultraprecision cutting with a single-crystal diamond tool to obtain a precise aspherical shape and diffractive pattern (Hirai, 1986; Yan et al., 2004; Yan, 2006). Under appropriate conditions Ni-P plated steel moulds can be successfully applied to the moulding of glass lenses (Masuda et al., 2007) and other microstructures (Yan et al., 2008). However, when the moulding temperature is very high, Ni-P plated moulds tend to cloud and roughen, and the moulded lens sometimes adheres to the surface of the mould. This phenomenon greatly reduces the practical service life of the moulds.

The present work is intended to clarify the causes of the cloud phenomenon of the moulds during the glass lens moulding processes. We investigated in detail changes in the subsurface elemental composition and surface topography of the mould during a heating process using various methods of characterisation, and revealed the atomic diffusion phenomena in the near surface layers of the moulds.

2 Experiments

2.1 Mould fabrication

To make observation easy, we used flat moulds instead of curved ones. The mould substrates are made of hot-work tool steel which remains relatively strong in high temperatures. After hardening, the substrates are then electroless-plated with Ni-10 wt%P (thickness 100 μm). Next, the plated surface is polished to a mirror finish. Finally, a release agent coating is applied. Generally, because the moulded glass sticks if the surface of the moulds has oxidised, the release agent coating is made of precious metal or Diamond Like Carbon (DLC) in order to prevent oxidation of the surface of mould (Kim et al., 2007; Ma et al., 2008; Kuribayashi et al., 1989). In the present experiment, an Ir-40 wt%Pt layer (thickness 300 nm) was sputtered as the release agent coating. Before the release agent coating, a thin Ni interlayer (thickness 50 nm) was coated onto the Ni-P plated layer to improve the adhering strength of the release agent coating. The structural diagram of the mould is given in Figure 1 and the sputtering

conditions in Table 1. For comparison, some moulds were specially made without Ni-P plating and some other moulds without the Ir-Pt layer and Ni interlayer.

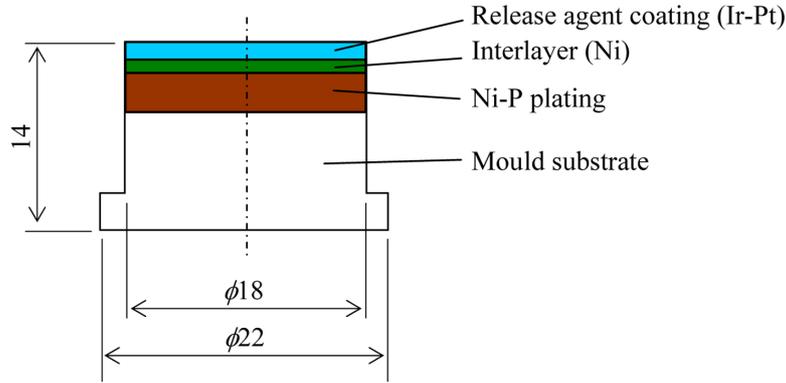**Figure 1**　Schematic diagram of mould structure (see online version for colours)

Table 1　Sputtering conditions

Materials	1st layer: Ni
	2nd layer: Ir-40wt%Pt
Thickness	1st layer: 50 nm
	2nd layer: 300 nm
Attainment pressure	5×10^{-4} Pa
Sputtering pressure	6.7×10^{-1} Pa with Ar gas
Electric power	RF 300 W
Substrate heating	300°C

2.2　Heating tests

In the present experiment, changes in composition and topography of the release agent coating were investigated by heating the moulds without pressing any glass. The moulds were heated in nitrogen at atmospheric pressure. Table 2 shows the heating conditions. Figure 2 shows photographs of two moulds made under the same conditions but heated at different temperatures. At a low temperature (400°C), even after heating for 32 h, the mould surface remained clear, as shown in Figure 2(a). However, after heating for 1 h at a high temperature (570°C), the mould surface was very cloudy, as shown in Figure 2(b).

Table 2　Heating conditions

Temperature	400, 500, 570°C
Atmosphere	Nitrogen
Pressure	1 atm
Time	1, 2, 4, 6, 8, 12, 16, 32 h

Figure 2   Photographs of moulds after being heated at different temperatures: (a) 400°C for 32 h
and (b) 570°C for 1 h (see online version for colours)

(a)   (b)
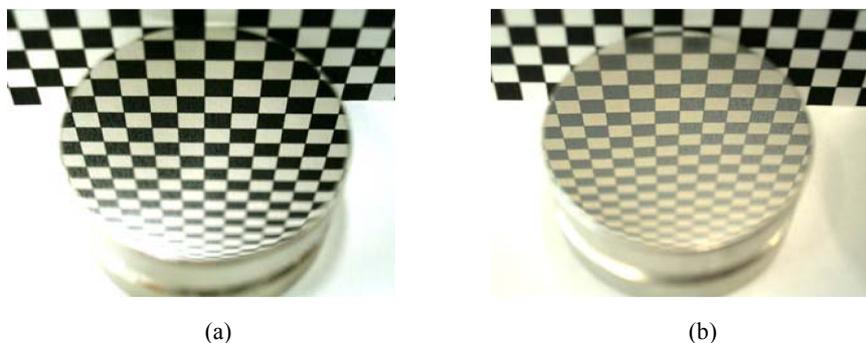
2.3   Surface characterisation

Before and after heating, surface roughness was measured with a surface roughness
measurement instrument Zygo NewView 5022, produced by Zygo Corp. In addition,
the topography of the surface was measured with an Atomic Force Microscope (AFM)
produced by SII NanoTechnology Inc. To examine mutual diffusion of the composition
elements between the plating layer and the release agent coating, various analyses were
carried out using Secondary Ion Mass Spectroscopy (SIMS) SIMS-6800T produced
by Physical Electronics Inc., an X-ray Photoelectron Spectroscopy (XPS) AXIS-HS
manufactured by Kratos Analytical Ltd., a Transmission Electron Microscope (TEM)
HF-2000 produced by Hitachi, Ltd., and an Energy Dispersive X-ray (EDX)
micro-analyser EMAX Energy EX-5770W produced by Horiba, Ltd.. The release agent
coating and interlayer were partially cut out using a Focused Ion Beam (FIB) processor
FB-2000 manufactured by Hitachi, Ltd. for cross-section observation.

3   Results and discussion

3.1   Changes in surface roughness

Figure 3 gives the relationship between heating time and surface roughness of moulds
that had an Ir-Pt sputtering layer, Ni interlayer and Ni-P plating layer at various
temperatures. Though surface roughness had not changed at 400°C, it did at 500°C and
570°C in accordance with the heating time. Surface roughness was greatly influenced
by the heating temperature. Surface roughness increased initially at high temperatures.
Figure 4 gives the results for a mould without Ni-P plating and another mould without
Ir-Pt layer and Ni interlayer. Surface roughness of the mould without the Ni-P plating
layer and that without Ir-Pt and Ni layers did not change even when heated at 570°C.
Therefore, we can say that the increase in surface roughness caused by heating
is a characteristic phenomenon for moulds that have Ir-Pt and Ni sputtering layers
on a Ni-P plating layer.

Figure 3 Plot of changes in surface roughness of the moulds with respect to heating time for the Ir-Pt/Ni/Ni-P/Steel mould (see online version for colours)
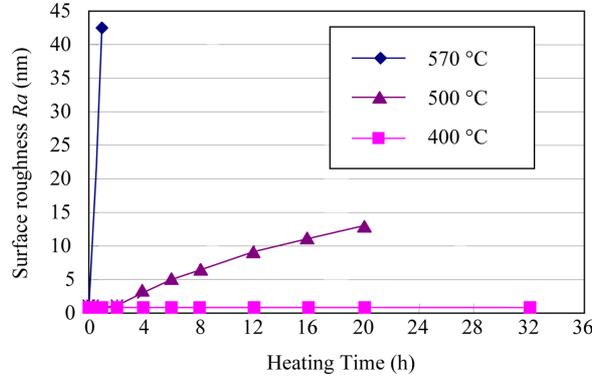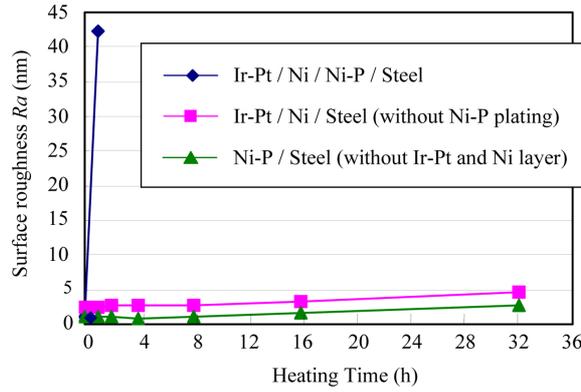

Figure 4 Influence of mould surface structure on surface roughness change of the mould during heating (see online version for colours)

The above results showed that the change in surface roughness is greatly influenced by the heating temperature. Therefore, it can be considered that a correlation exists between the diffusion of the atoms in each layer and the increase in surface roughness. The diffusion coefficient D, which is the index of the ease at which the atoms diffuse, is given using equation (1) (Sudo et al., 1972).

$$D = D_0 \exp(-Q/RT) \tag{1}$$

where D_0 is the frequency factor, Q is the activation energy of diffusion, R is the gas constant, and T is the temperature in Kelvin. Generally, the frequency factor D_0 ranges from $0.1 \text{ cm}^2\text{s}^{-1}$ to $10 \text{ cm}^2\text{s}^{-1}$, and the activation energy of diffusion Q in the case of boundary diffusion is approximated using $10RT_m$ (T_m: melting temperature in Kelvin) for metals (Cottrell, 1969). The relation between the diffusion coefficient D and diffusion distance of atom L is given using equation (2) (Sudo et al., 1972).

$$L = (2Dt)^{1/2} \tag{2}$$

where t is the time.

The relationship between surface roughness and the square root of the heating time in the case of being heated at 500°C is given in Figure 5. After 2 h of heating, the surface

roughness is proportional to the square root of the heating time. This proportional relationship between surface roughness and heating time is similar to that between the diffusion distance of atoms and the heating time given by equation (2), thereby leading to the consideration that the increase in surface roughness is closely related to the diffusion of the atoms.

Figure 5   Relationship between surface roughness and heating time when heated at a temperature of 500°C (see online version for colours)
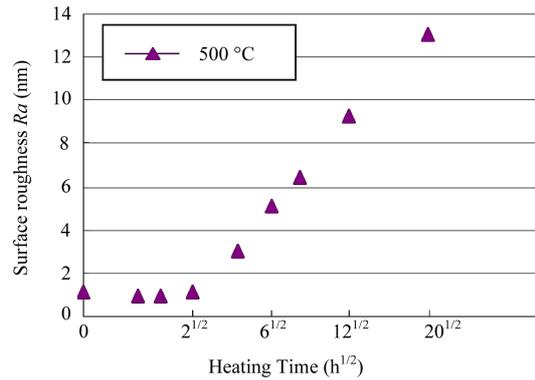
However, the 2 h from the beginning of heating, required for Ni atoms to reach the surface from beneath the release agent coating, also needs to be taken into consideration. If the thickness of the Ir-40wt%Pt layer is 300 nm and the elapsed time for the Ni atoms to arrive at the surface is 7200 s, which are then used in equation (2), the experimental diffusion coefficient D would be 6×10^{-14} cm^2/s. If in equation (1), a melting temperature of Ir-40wt%Pt layer T_m of 2446 K is used, which was approximated using the melting temperatures of Ir and Pt, a frequency factor D_0 of 0.1–10 cm^{2}s^{-1}, the activation energy when Ni atoms diffuse through a crystal boundary is assumed to be $Q = 10RT_m$, the calculated diffusion coefficient of Ni into the Ir-40wt%Pt layer would be $2 \times 10^{-13} \sim 2 \times 10^{-15}$ cm^2/s. This calculated diffusion coefficient is similar to the experimental diffusion coefficient, and hence it can be confirmed that the increase in surface roughness of the mould is influenced by the diffusion of Ni atoms from beneath the release agent coating.

3.2   Changes in subsurface composition distribution

The depth profile of each element from the surface of the mould was measured with SIMS analysis. The depth profile of the mould which had not been heated is given in Figure 6, where the Ir-Pt release agent coating (300 nm in thickness) and the Ni interlayer (50 nm in thickness) are formed in accordance with the design. On the other hand, the depth profile of the mould heated for 1 h at 570°C is shown in Figure 7. In this case, however, mutual atomic diffusion among the release agent coating, the interlayer and the Ni-P plating layer occurs. Ni atoms diffuse to the surface of the release agent coating, and the Ni concentration gradient from the release agent coating to the plating layer is almost lost. With regard to the element P, the secondary ion strength rises remarkably in the release agent coating area compared with Figure 6. Therefore, we can say that P diffuses into the release agent coating from the Ni-P plating layer too. Due to the

external diffusion of P, the P concentration in the outer plating layer of depth range 0.3~0.8 µm has been lowered significantly. Concurrently with the external diffusion of Ni and P, Pt and Ir diffuse internally from the release agent coating to the Ni-P plated layer. As a result, the concentrations of Pt and Ir on the outer surface decrease distinctly.

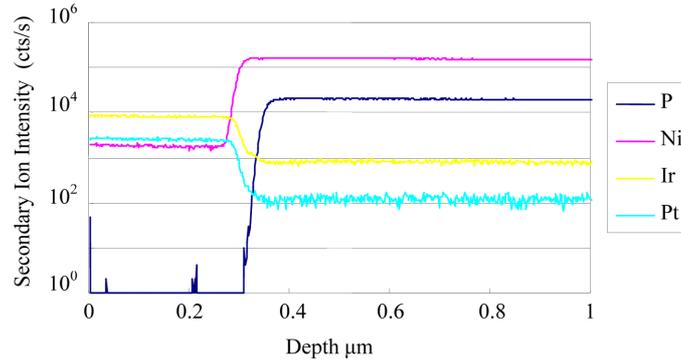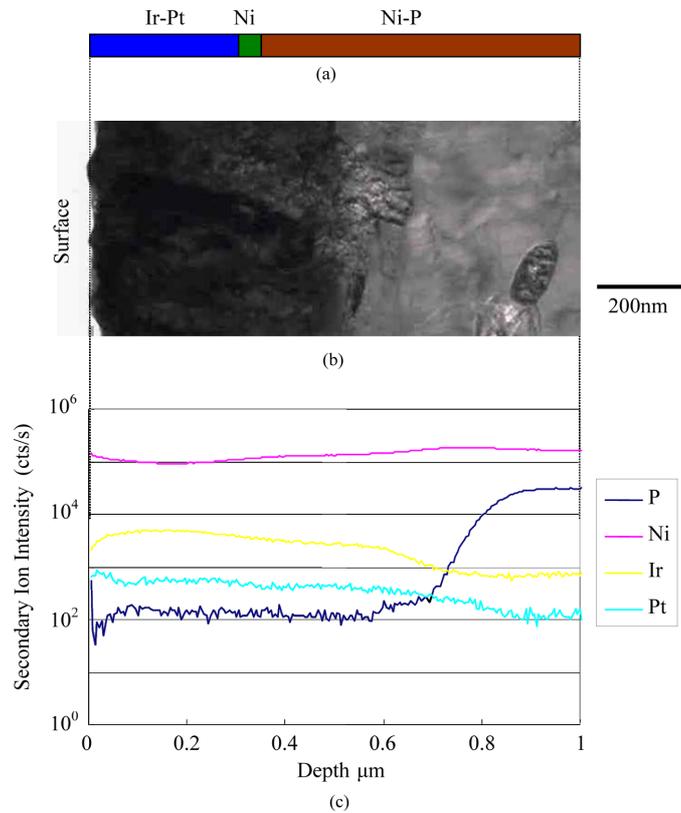**Figure 6**   Depth profile of element distribution in Ir-Pt/Ni/Ni-P/Steel mould before heating (see online version for colours)

Figure 7   Cross-sectional photograph and depth profile of the mould composition: (a) schematic diagram before heating; (b) TEM image and (c) depth profile after heating at 570°C for 1 h (see online version for colours)

From the cross-sectional TEM photograph in Figure 7(b), we can see that the boundary between the release agent coating and the interlayer is very indistinct because of their mutual diffusion. The boundary of the Ni-P plating is in a position about 700 nm deep from the surface. This boundary, as can be seen in the TEM photograph, basically corresponds to the range of mutual diffusion shown in the SIMS result in Figure 7(c).

It is noticed that there is a peak on the secondary ion intensity curve of P at the mould surface after heating in Figure 7(c). A similar phenomenon can be confirmed with the result before heating in Figure 6, too. To confirm the presence/absence of P in the surface of the mould, a mould heated for 1 h at 570°C was investigated with XPS. The result of XPS analysis is given in Table 3, where no P was detected. From these results, we presume that the peak of P seen in the surface when performing SIMS test might be a kind of measurement error.

Table 3   Surface composition of the mould surface measured by XPS after heating at 570°C for 1 h

Ir	Pt	Ni	P (wt%)
17.28	31.03	51.69	0

## 3.3   Changes in surface topography of the mould

The surface topography of the mould before and after heating at 570°C for 1 h was observed with AFM, and the results (top views and cross-sectional profiles) are given in Figure 8(a) and (b), respectively. Although the surface of the mould before heating was smooth, several swellings of about 3 μm in diameter and about 200 nm in height can be seen all over the mould after heating. It is thought that the partial swelling on the mould surface is caused by mutual atomic diffusion between the release agent coating layer, the interlayer and the plating layer. It is these partial swellings of the surface that caused the cloudiness of the mould after heating.

Figure 8   Surface topography of the Ir-Pt/Ni/Ni-P/Steel mould measured by AFM: (a) before heating and (b) after heating at 570°C for 1 h (see online version for colours)

(a)   (b)
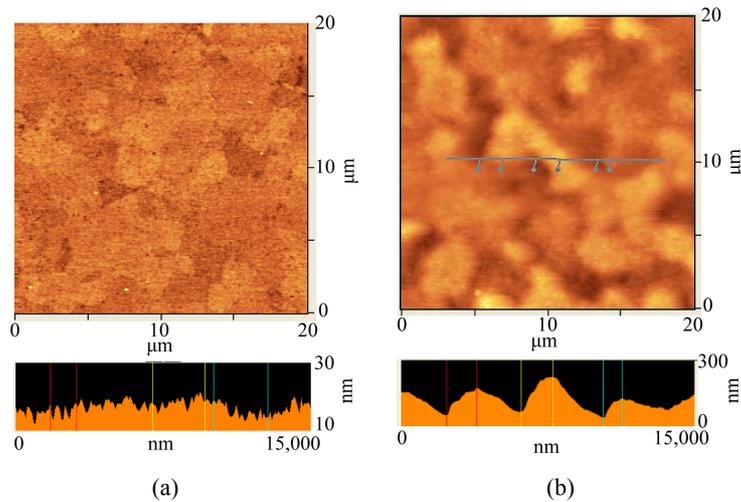
SIM photographs of the swelling regions after heating are given in Figure 9. Figure 9(a) is a photograph of swelling region on the mould surface; Figure 9(b) and (c) are photographs of a cross section of the swelling region taken at different magnifications. The cross section sample was cut out using FIB etching, and observed from 60° on the diagonal. It can be seen that the release agent coating and interlayer have become integrated through mutual diffusion. The interface with the plating layer is located at a depth of about 700 nm from the surface, and corresponds to the graph of the concentration distribution of SIMS given in Figure 7(c). It is noticeable in Figure 9(c) that the interface with the plating layer is undulating and corresponds to the shape of the surface swelling. From this result, it is thought that the swelling of the surface is influenced by atomic diffusion from the plating layer.

Figure 9   SIM photographs of mould surface after heating: (a) a swelling part; (b) cross section of a swelling after ion etching and (c) magnified view of (b) showing undulation of interface (see online version for colours)
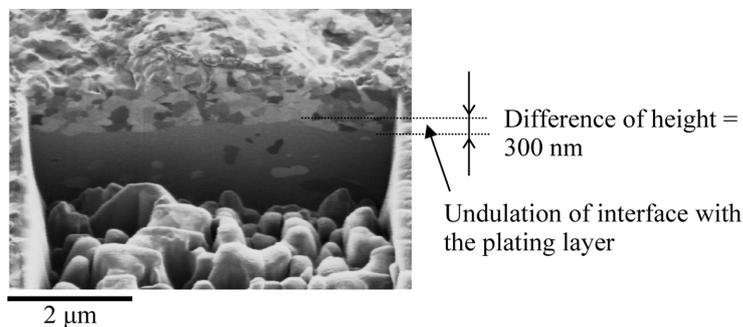

5 μm

(a)
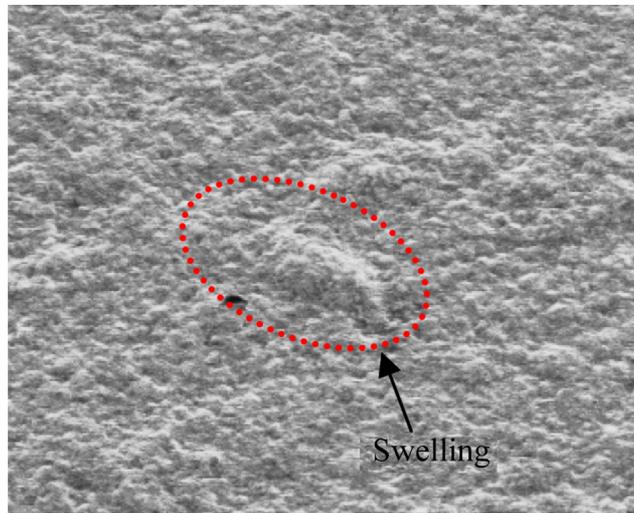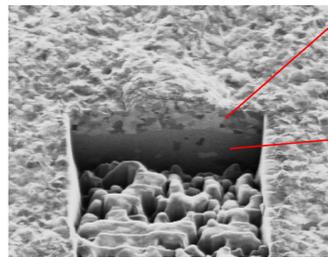
Release agent coating (Ir-Pt) layer integrated with interlayer (Ni)

Ni-P layer

3.5 μm

(b)

Figure 9   SIM photographs of mould surface after heating: (a) a swelling part; (b) cross section
of a swelling after ion etching and (c) magnified view of (b) showing undulation of
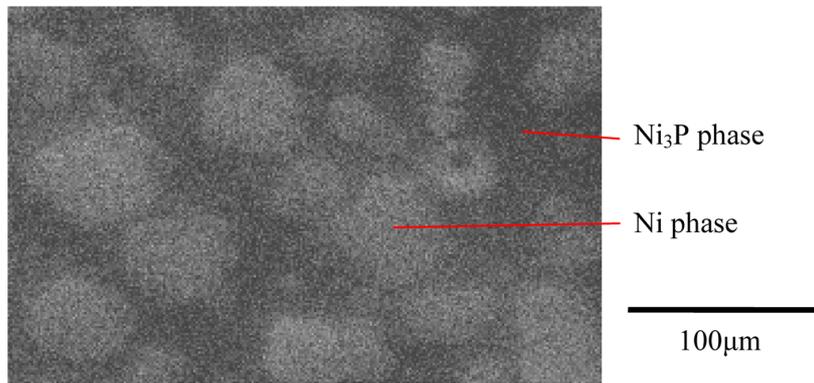interface (see online version for colours) (continued)

(c)

The cause of the swelling of the mould surface is, presumably, that a large amount
of Ni diffuses from the plating layer to the release agent coating after heating. According
to the Kirkendall effect (Cottrell, 1969), the number of Ni atoms diffusing from
the plating layer to the surface of the mould would be greater than the number of Pt and
Ir atoms in the release agent coating that diffuse internally. If the Ni atoms diffused
uniformly throughout the mould surface, however, partial swelling would not occur.
The fact that the surface swells partially demonstrates that the rate of diffusion of Ni
atoms differs from place to place.

Because the structure of the Ni-P plating is usually composed of two phases: Ni and
Ni_3P, in the next step, the difference in the diffusion rate of Ni atoms in the Ni phase and
in the Ni_3P phase was investigated. In order to reveal the correlation between the partial
swelling and the two phase structure of the plating, a Ni-P plated mould was first heated
at a high temperature 920°C before sputtering the Ni interlayer and the Ir-40wt%Pt layer.
At this high temperature, crystal grains in the Ni-P plating layer can easily grow to very
big, which facilitate experimental observation. After heating, the mould surface was
polished and observed. The EDX mappings of the Ni-P plating obtained in different
modes are given in Figure 10. It is clear that the structure of the Ni-P layer is composed
of Ni crystal grains (bright regions in Figure 10(a) and dark regions in Figure 10(c))
precipitated in the Ni_3P matrix.

Subsequently, a Ni interlayer and an Ir-40wt%Pt layer were sputtered onto this
mould with the sputtering conditions shown in Table 1; thus, the mould structure became
the same as that shown in Figure 1. Then, the sputtered mould was again used for heating
tests at 570°C for 1 h. The topographies of the mould surface before and after heating are
shown in Figures 11 and 12, respectively. Before heating, as shown in Figure 11, the
maximum height difference is 46 nm; while after heating, the maximum height difference
increases to 288 nm. Also, it is clear that the swellings occur in the Ni_3P phase regions
surrounding the Ni crystal grains. This fact suggested that the external Ni diffusion
occurs faster for the Ni_3P phase than for the Ni phase in the Ni-P plated layer. The release
agent coating swells up due to the inflow of a large amount of Ni atoms from the Ni-P
layer. The diffusion rate of the Ni differs from point to point; thus, the surface of the
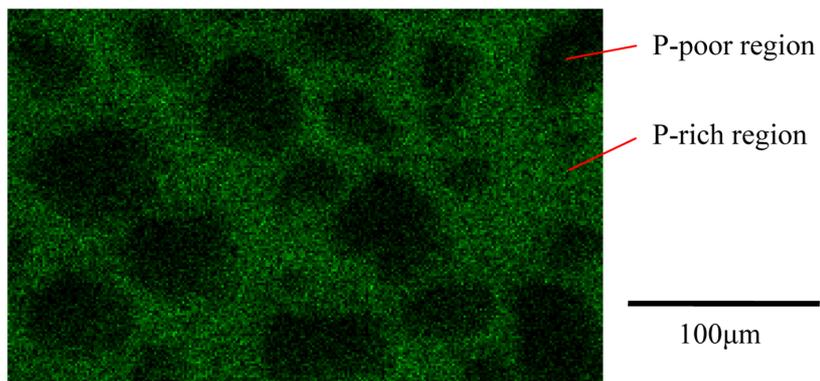mould is roughened.

Figure 10 EDX mapping of the Ni-P plating heated at 920°C: (a) secondary electron image; (b) Ni distribution and (c) P distribution (see online version for colours)
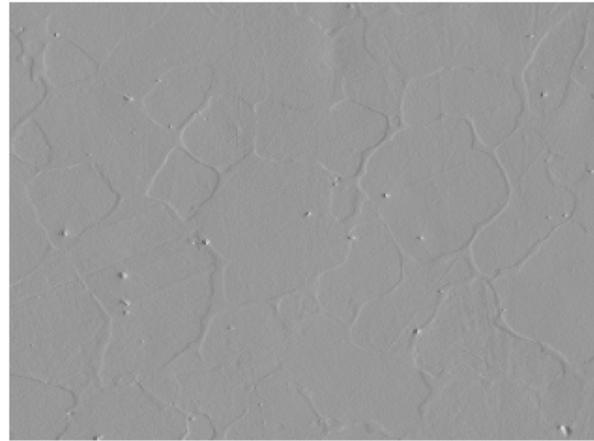

Ni$_3$P phase

Ni phase

100μm

(a)
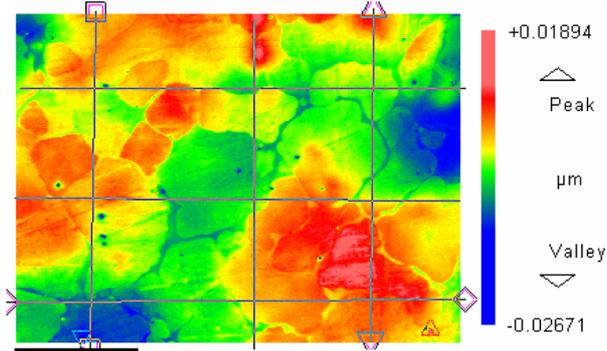

100μm

(b)

P-poor region

P-rich region

100μm
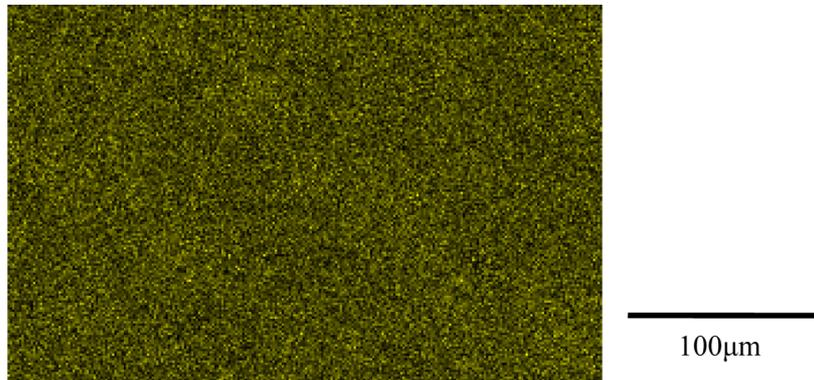
(c)

Figure 11 Surface topography of the mould with large Ni-P crystal grains before heating:
(a) Optical microscope image; (b) height distribution in plane view and (c) height
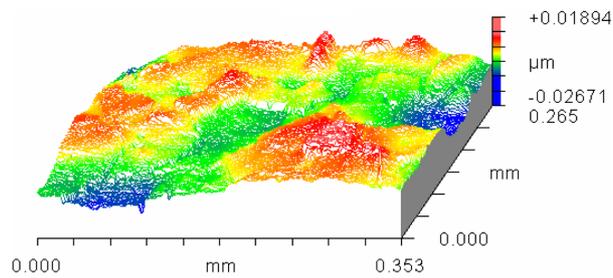distribution in bird's-eye view (see online version for colours)

100μm

(a)

100μm

(b)

The maximum difference of height = 46 nm

(c)

Figure 12 Surface topography of the mould with large Ni-P crystal grains after heating at 570°C
for 1 h: (a) optical microscope image; (b) height distribution in plan view and
(c) height distribution in bird's-eye view (see online version for colours)
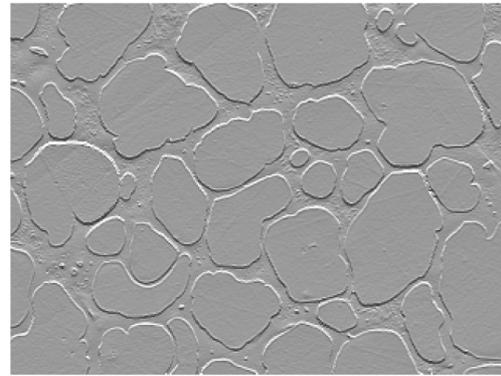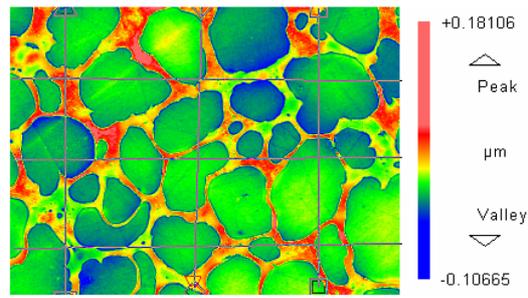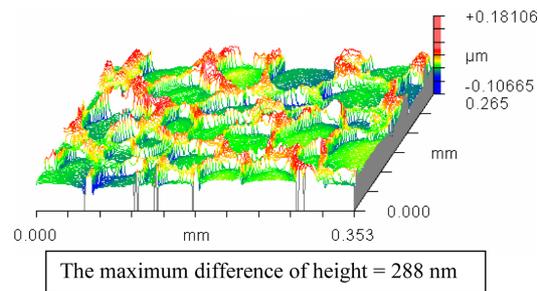

100μm

(a)

100μm

(b)

The maximum difference of height = 288 nm

(c)

It is also noticeable from Figure 4 that the fast diffusion of Ni atoms does not occur in moulds without Ni interlayers and Ir-Pt layers. Therefore, we may presume that these sputtered layers influence the Ni diffusion from the Ni-P layer. When a thin film of the different material is laminated and heated, the interface of each thin film may get highly stressed because of the differences in crystal structures, lattice constant, and thermal expansion coefficient (Miura, 1995). The developed stress easily concentrates at the

crystal grain boundaries (Miyamoto et al., 1971), and when the stress localises, the diffusion of the atoms is influenced by that stress. In this case, the diffusion coefficient D is given by equation (3) (Kao et al., 1988).

$$D = D_0 \exp(-Q/kT) \exp(-\sigma V_a/kT) \tag{3}$$

where D_0 is the frequency factor, Q is the activation energy of diffusion, k is the Boltzmann constant, T the temperature in Kelvin, σ the stress, and V_a the constant known as the activation volume and is a concept for revealing the area where the stress works. The Boltzmann constant k is a value in which the gas constant (R) is divided by the Avogadro constant (N_A), as shown in equation (4).

$$k = R/N_A. \tag{4}$$

If a high compression stress works on the interface of the Ni-P layer where the thermal expansion coefficient is larger than the Ir-Pt layers during heating and the stress concentrated at the grain boundaries, the diffusion of Ni atoms will occur easily at the grain boundary between Ni and Ni_3P. The swelling of the release agent coating in the present study occurred to the Ni_3P phases of the Ni-P layer; hence, this phenomenon might be related to the stress concentration at the interface between the Ni_3P phase and the Ni phases in both the plating layer and the interlayer. A schematic model of the atomic diffusions among the near surface layers of the mould is given in Figure 13.

Figure 13 Schematic model of atomic diffusions among the surface layers of the mould
(see online version for colours)
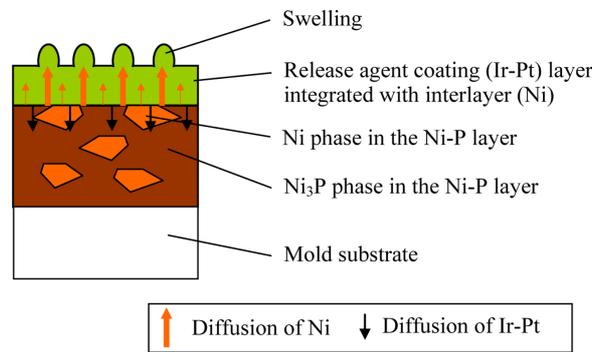
It is also noteworthy that although a combination of a similar interlayer and a release agent coating is also used in moulds made of cemented carbide, the cloudy phenomenon on the mould does not occur at a high temperature of about 600°C. The reason for this is thought to be that the amount of atomic inflow from the cemented carbide to the release agent coating through diffusion is small. Although Ni atoms in the interlayer can diffuse too, the amount of Ni atoms is not enough to swell the release agent coating. In the case that the interlayer and release agent coating were sputtered on the steel substrate without Ni-P plating, the cloudy phenomenon was not seen, as given in Figure 4. From these results we concluded that the cloudy phenomenon on Ni-P plated moulds occurs when there are sufficient Ni atoms in the Ni-P plating which can flow into the release agent coating. Therefore, to prevent the cloudy phenomenon occurring on Ni-P plated moulds, an effective way would be to restrict the diffusion of Ni atoms into the release agent coating.

4   Conclusions

To clarify the causes of the cloudy phenomenon of Ni-P plated moulds occurring in the glass lens moulding process, the changes in topography and material element distribution of the mould surface caused by heating were investigated. The conclusions can be summarised as follows:

- The cloudy phenomenon of a mould is a result of the increase in surface roughness. The higher the temperature, the rougher the mould surface.

- In a cloudy mould, Ni in the Ni-P plating layer diffuses to the outer layer of the release agent coating. At the same time, Ir and Pt in the release agent coating diffuse in a reverse direction into the inner Ni-P plating layer.

- The diffusion speed of Ni is higher than that of Ir and Pt, causing the interface between the release agent coating, and the Ni-P plating layer shift towards the mould substrate.

- In the swelling part of the mould surface, the interface between the Ni-P plated layer and the release agent coating shows undulations corresponding to the surface topography, indicating that the diffusion speed and amount of atoms are location-dependent.

- Surface regions corresponding to the locations of Ni_3P phases in the Ni-P plating are significantly higher than those corresponding to the Ni grains. Ni atoms in the Ni_3P phases flow copiously into the release agent coating, swelling the mould surface.

In the next step we will further investigate the stress state at the interface between the release agent coating and the Ni-P plating and its effects on the diffusion of Ni atoms. We will also explore new subsurface structures for the moulds for restricting atomic diffusion.

Acknowledgements

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