Reversal Ag Stain with Reduction Fogged AgCl Emulsion on Different UV Energies and Exposure Times

Tai Sung Kang and Seog Jung Lee
Research Center of Hanmi Filmtech Co., Ltd., Chungbuk, Korea

Abstract

We made silver chloride cubic crystal emulsion in size of 800-1000 nm and then added reduction agent (fogging agent) to have reduction fogged AgCl into which we added with UV absorber, desensitizer and other additives to coat it on the PET film in thickness of 5. We had the coated film samples exposed to the ultra violet (260-380 nm) on different unit mJ/cm² and different exposure time so as to study reversal Ag stain which were developed on film samples.

Introduction

Generally, manufacturing process of direct reversal emulsion is subject to the indication of the product and some photographic elements are being considered. The silver halide emulsion for the direct positive silver halide photographic light sensitive material is fogged in advance and Herschel effect is utilized to break fogging nuclear by exposure, whereby a positive image is formed.

In direct positive emulsion, silver halide is reduced by reducing agent so as to make a surface latent image which is then exposed to the light to form Ag nucleus and fogged.

The fogging reducing agent useful for preparing emulsion include thiourea dioxide, hydrazine compound, polyamine compound, amine borne, stannouschloride, tin(II) chloride, the metal compound which is electrically more positive than silver included, soluble salt of rhodium, iridium, gold and palladium.

The initial process of direct reversal emulsion is to make a shape of silver halide particle and it is well known that the method requires some chemicoophysical elements for the reaction of silver nitrate and salt halide such as pH, pAg, time and temperature, etc.

The second process is to reduce Dmin meanwhile making a surface latent image on the silver halide particle which was reduced by reducing agent.

The third is to get Dmax, low sensitivity and log fog after reversal in the process of internal latent image which was doped on silver halide with a heavy metal.

There have been a lot of studies on the photochemical and physical effects of Rhodium salt and Iridium salt on the photographic emulsion doping.

In this study, we used Rhodium salt and Iridium salt to make reduction fogged AgCl emulsion. Reversal Ag stains formed on different UV energies and exposure times will be discussed. In the previously mentioned process, controlling skills of photographic properties by desensitizer and stabilizer, other than heavy metals might be required.

Experiment

In this study, to make particles, we mixed 10% NaCl of 5ml (solution A) in 10% inert gelatin 1000ml and used double-jet method for injecting AgNO₃ of 1M (solution B) and NaCl of 1.14M (solution C) at the temperature 62°C for 55 minutes under the conditions of pH4.8 and pAg 7.1 and then cooled them down to 30°C and get 100% AgCl cubic crystal in the range of 800nm-1000nm.

After washing, we tested Dmin on 5.09×10⁻¹⁰ mol K₂RhCl₆ /mol Ag, 5.09×10⁻¹⁰ mol K₂IrCl₆ /mol Ag and mixture of the two (2.5×10⁻¹⁰ mol K₂RhCl₆ /mol Ag + 2.5×10⁻¹⁰ mol K₂IrCl₆ /mol Ag) and put 5.1×10⁻³ mol thiourea dioxide/mol Ag into all the three solutions.

Each testing AgCl emulsion was coated on a PET base at coating weight of 4.0g AgX /m² and thickness of 5 μm. We gave tungsten light source and UV to study the photographic properties and direct reversal fogged stain.

Sensitometric: Nalumisensitemeter(Japan) 2850K 1/20sec.
UV Exposure (Korea): 260-380nm
Developer: 741KGD(Konica)-23°C (30sec.)
Fixer: 911KGF(Konica)-23°C (30sec.)

Result

From the experiment of putting water soluble metal and surface developing, we got a result as shown in the figure 1; higher Dmin and sensitivity from the mixture.
of $K_2\text{RhCl}_6 + K_2\text{IrCl}_6$ than from each $K_2\text{RhCl}_6$ or $K_2\text{IrCl}_6$.

When we put thiourea dioxide into three solutions above as shown the table 1, we found that Dmax of $K_2\text{RhCl}_6$, $K_2\text{IrCl}_6$ were improved meanwhile sensitivity of the two were decreased and that complexes of $K_2\text{RhCl}_6$ and $K_2\text{IrCl}_6$ had low Dmax and fast sensitivity.

![Figure 1](image1.png)

*Fig. 1 Photographic properties of additives consisting of $5.09 \times 10^{-10}$ mol $K_2\text{RhCl}_6$/mol Ag, $5.09 \times 10^{-10}$ mol $K_2\text{IrCl}_6$/mol Ag, $2.5 \times 10^{-10}$ mol $K_2\text{RhCl}_6$/mol Ag + $2.5 \times 10^{-10}$ mol $K_2\text{IrCl}_6$/mol Ag for 1/20sec. exposure*

Table 1. Photographic properties after adding thiourea dioxide

<table>
<thead>
<tr>
<th></th>
<th>$K_2\text{RhCl}_6$</th>
<th>$K_2\text{IrCl}_6$</th>
<th>$K_2\text{RhCl}_6$, $K_2\text{IrCl}_6$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dmax</td>
<td>4.0</td>
<td>4.5</td>
<td>4.2</td>
</tr>
<tr>
<td>Sensitivity</td>
<td>34</td>
<td>30</td>
<td>38</td>
</tr>
</tbody>
</table>

Figure 2 is a 500 times picture of the reversal fogged area/cm$^2$ after developing and fixing upon the different UV exposures.

In case of $K_2\text{RhCl}_6$, as shown in the figure 2, both too much or too less UV exposure made fogged stain in the sample meanwhile, in case of $K_2\text{IrCl}_6$, we found that the higher UV exposure, the more apparent fogged stain and that the lower UV exposure, the less fogged stain in the sample.

In case of the mixture of $K_2\text{RhCl}_6+K_2\text{IrCl}_6$, just as the same in case of $K_2\text{RhCl}_6$ alone, we found some fogged stain remained. This result was the same as shown in the table 1, fogged stain remained under the conditions of low Dmin, fast sensitivity, and the changes in UV exposure after reversal fogged development.

![Figure 2-1](image2.png)

*Fig. 2-1 Fogged stain upon UV exposure of doping sample ($5.09 \times 10^{-10}$ mol $K_2\text{RhCl}_6$/mol Ag)*
Figure 2-2 Fogged stain upon UV exposure of doping sample (5.09×10^-10 mol K2IrCl6/mol Ag)  

Figure 2-3 Fogged stain upon UV exposure of doping sample (2.5×10^-10 mol K2RhCl6/mol Ag + 2.5×10^-10 mol K2IrCl6/mol Ag)  

Figure 3 shows that the number of fogged stain was not decreased in K2RhCl6 and K2RhCl6+K2IrCl6 even through the changes in UV exposure, but in case of K2IrCl6, we didn’t find a fogged stain at the range of 4.5mJ/cm² ~ 5mJ/cm².  

Conclusion  

Through the test of coating film with reduction fogged AgCl emulsion upon different UV energies and UV exposure times, we found fogged stain remain from the doping-desensitized emulsions of K2RhCl6 and from the complex of K2RhCl6+K2IrCl6 even after forming Ag fogged by thiourea dioxide and the changes in UV energies and exposure times. However, in case of K2IrCl6, we did not find any fogged stain remain at the UV energy of 5mJ/cm².  

From this result we learned of a relation which remaining of fogged stain would have, in the process of direct reversal, with the volumes as well as kinds of metal dopants in nano-size cubic grain of AgCl emulsion and with the volumes of fogged agent and with the different UV energies and exposure times.
Reference

1. T.H. Jame, The theory of the photographic process, Third P.149

Biographies

Tai-Sung. Kang obtained Ph. D at ChungAng University, Korea in 1984. He had studied on AgX for more than 30years at the Korea Research Institute Chemical Technology. Since 1998, he has been the chief of R&D Center of Hanmi Film tech Company and has been directing studies and projects on inkjet printing media.

Seog-Jung. Lee received Mas. at SoongChunHyang University, Korea in 2003. Since 2003, he has been researcher of R&D Center of Hanmi Film Tech Company and studied photographic materials.