How to identify Douros synthetic rubies

A new synthetic ruby called Douros, produced by a flux process in Greece, is being sold worldwide and polished is difficult to identify using standard gemmological tests.

Picture one: Douros synthetic rubies, the largest stone is 4.20 carats

Picture two: most of the crystals possess a rhombohedral shape, upper right. A typical tabular crystal with coarse flux inclusion is on the lower left, and a cluster is shown in the upper left. Largest crystal is 3cm across

Since the production of Ramaura synthetic rubies by J. O. Crystal Co Inc in the United States, the gemmological community and the trade have become aware of synthetics that are difficult to identify.

Identification of modern synthetic stones seems to be more and more a laboratory matter, since, in an increasing number of cases standard gemmological methods can no longer reveal the difference between natural and artificially grown crystals.

In April 1993 a new synthetic ruby called Douros, grown by a flux process, was tested independently by two gemmological laboratories in Switzerland.

Identification of clean or slightly included stones may create considerable difficulties if standard gemmological tests are performed alone. Even when advanced tests are applied, such as UV-VIS spectrophotometry, energy dispersive X-ray fluorescence, or identification of growth planes, the material may cause difficulties in identification.

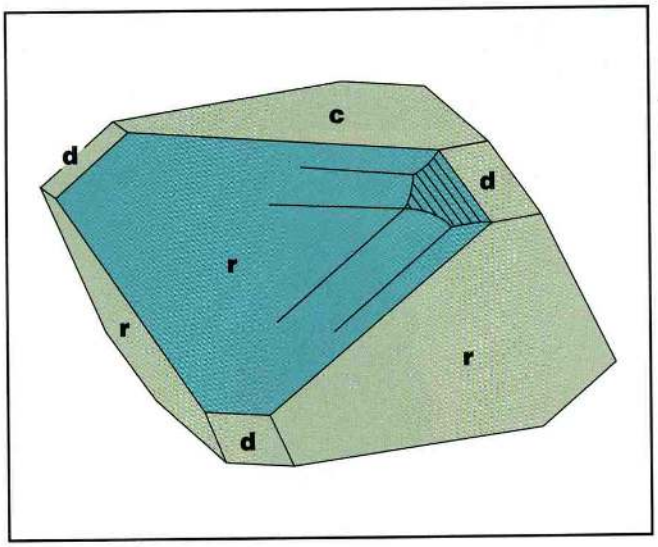
Producers of the synthetics are J. & A. Douros, a small family business owned by two brothers, John and Angelos Douros, in Piraeus, Greece. One of the owners is a physicist and the other an electrical engineer and the company earlier specialized in precious metal refining.

Using self-built furnaces they produce synthetic rubies by a flux technique, with controlled spontaneous nucleation. Seed crystals are not used. After some years of experiments they started commercial production in 1993. The stones were shown at Athens Jewellery Fair for the first time in February 1993.

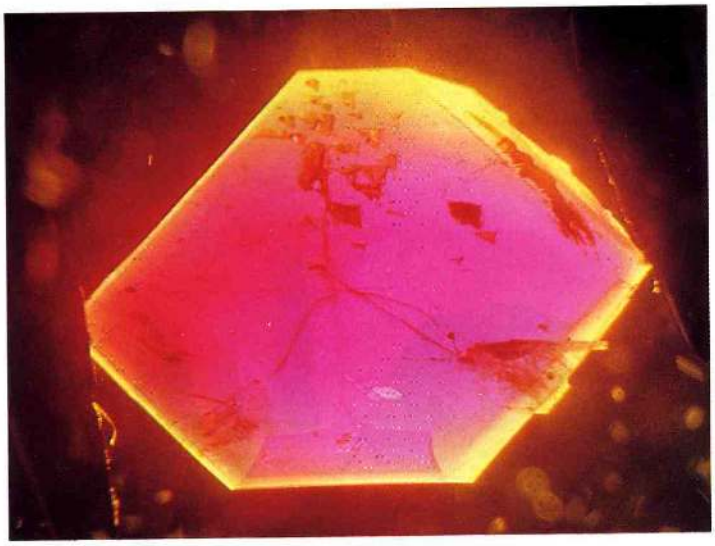
The goal of the producers is to make their synthetic rubies as close as possible to natural. They use various trace elements to obtain different shades of colour seen in natural

continued on page 160

By Dr Henry Hanni, director of Swiss Gemmological Institute, or SSEF, in Zurich, Switzerland and Dr Karl Schmetzer in Petershausen, Germany



Picture three: schematic sketch of a rhombohedral crystal, dominated by positive and negative rhombohedral faces r, the basal plane c and a small face d. On the sketch, the formation of the umbrella is shown by the growing d-plane sector. The d plane is visible as a small shinning rectangle in picture two, upper right



Picture four: crystal immersed in dijodmethane, exhibiting the faces r and d in conjunction with the characteristic umbrella pattern, representing the traces of the growing d face at the lower end of the crystal. On the top of the crystal, residual flux particles are visible. Note the colourless rim of the crystal

rubies. The individual growth technique results in higher production costs compared to crystals produced by the Verneuil or Czochralski pulling method.

The Douros flux method may yield crystals of 20 to 50 grams and smaller. The largest is a 70-gram crystal. Monthly rough production is 2,000 carats. Crystals are sold to a few dealers in different countries who reportedly appreciate the good quality of these synthetics.

Colour of crystals varies from a saturated red to a violet-red and purplish-red, according to the amounts of chromium, titanium and iron present, **picture one**.

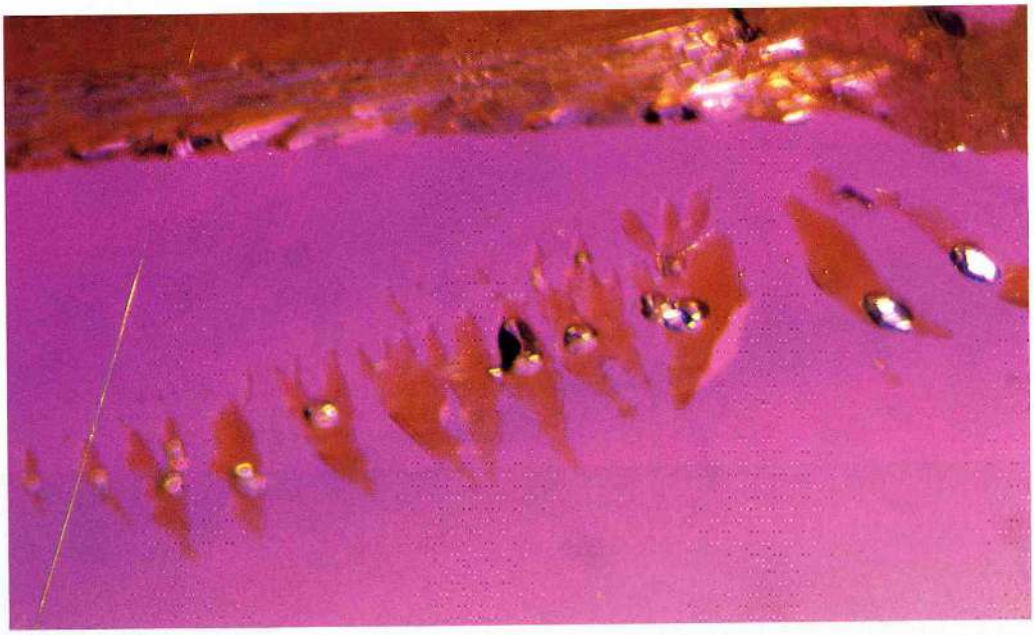
The rubies basically occur in three shapes: tabular crystals, rhombohedral crystals and clusters.

In some samples blue colour zones, representing a portion of blue sapphire, have been seen. Most of the synthetic rubies are isometric rhombohedral crystals with dominant basal c and rhombohedral r planes, sometimes with a subordinate negative rhombohedal d plane, **picture two**.

On the flat tabular crystals, dominated by basal c planes, interpenetrating or repeated twinning was observed, **picture three**. Most of the crystals show a colourless outermost layer which indicates the absence of chromium during the final growth period.

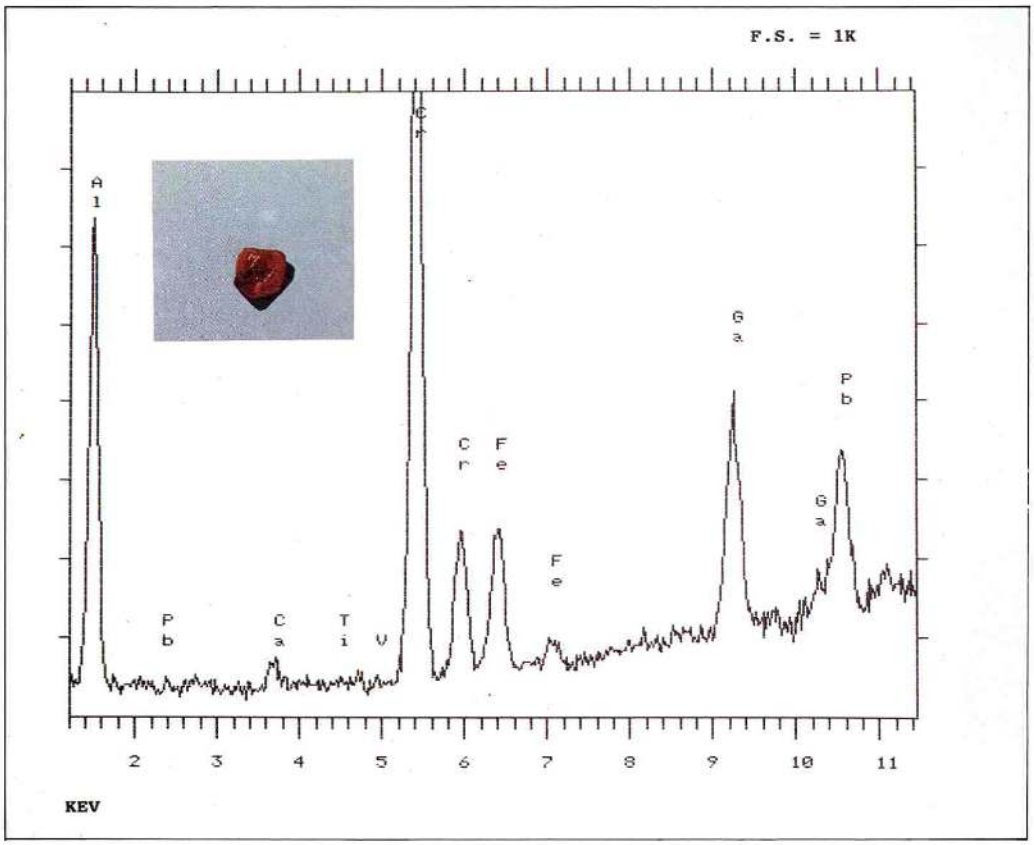
A definite colour zoning is observed with chromium and iron incorporated selectively in different growth directions, and are confined to different crystal faces. This may re-

continued on page 164



Picture five: small residual flux droplets with silver vacuum bubbles. The flux shows the characteristic colour of this lead compound

Picture six: the veil type of inclusions looks very similar to patterns seen in heat and borax-treated natural ruby with their droplets and network of intercommunicating channels. From a faceted Douros synthetic ruby



Picture seven: energy-dispersive X-ray fluorescence spectrum, giving qualitative evidence of the trace elements, chromium, iron, gallium and lead, the latter stemming from flux traces present in the analysed sample, a rhombohedral synthetic Douros ruby

sult in a peculiar growth pattern, described as umbrella-shape growth sector. The characteristic was earlier seen in Ramaura synthetic rubies.

Identification

Physical properties of the 12 Douros synthetic rubies investigated do not differ from natural stones in respect of refractive indices, density, absorption spectrum, fluorescence and dichroism.

Usually they are slightly included and do not have platinum platelets or lancets. Some showed residual flux droplets of an orange-yellow colour. The flux in larger portions is poly-crystalline and crazed in appearance, and usually contains a gas bubble.

Where the droplets are small, the yellow colour is no longer visible but the tiny bubbles are still present, **picture five**.

Such inclusions look very similar to heat and borax-treated natural ruby, which at present represents the bulk of natural rubies traded.

Fine stringers or dust-tracks are sometimes visible in dark field illumination which resemble the traces of meteorites. The composition of the flux was investigated by scanning electron microscope-Energy Dispersive System and was found to consist of a lead compound. In contrast to Ramaura synthetic rubies, bismuth and lanthane have not been found up to now. Although the rough crystals look almost identical to the Ramaura material, the flux

continued on page 168

composition is different.

The common characteristic structural feature for Ramaura as well as for the new Douros synthetic rubies seems to be the presence of a small d crystal face, **picture four**. It is related with the peculiar growth pattern, described as umbrella, which is formed by a d face, with neighbouring c faces and r faces. The observation of this peculiarity, usually visible only in immersion, is one of the safe means of identification although this pattern is confined to the outer zone of the crystal. It may often be taken away by cutting.

Chemical investigation by energy dispersive X-ray fluorescence revealed most of the trace elements seen in natural rubies. However, thorough comparison of titanium, vanadium, chromium, iron and gallium contents enables differentiation. Some lead has been identified by energy dispersive X-ray fluorescence which indicates the growth medium of the crystals.

Chemical composition

The chemical composition of five Douros samples was studied with microprobe technique. Aluminium-oxide content was found to be between 98.5 percent and 100 percent of weight. The remainder was substitution of aluminium by mainly colour-causing trace elements. Chromium was found to be up to 1.4 percent and iron was up to 0.3 percent of weight. Vanadium and manganese were found to be below detection limits of the microprobe. A significant zoning in chemical composition regarding chromium and iron has been observed.

Differentiation of natural ruby from Douros

Natural rubies are usually different from the new synthetics because of their structural characteristics and mineral inclusions. Natural rubies often show intercalated fine twin lamellae in one, two or three spacial directions, leaving the characteristic intersection lines. Such structural features were not observed in the Douros material, although a special type of repeated or interpenetration twinning was observed on crystals, as is occasionally seen in other types of flux synthetic rubies, **picture three**.

Even after heat treatment, included foreign crystals may exhibit their proper crystal shapes and help to identify natural ruby. Rutile needles, or traces after heat treatment, are not present in Douros synthetics.

If internal growth planes are studied by immersion microscopy, natural rubies usually show preferences for individual crystal faces which are not yet observed in synthetic flux grown rubies. On the other hand, most synthetic crystals possess growth planes which do not occur on natural material, for instance the umbrella pattern.