In commercial practice 5.5Ni steel is toughened for cryogenic service by a three-step heat treatment designated the "QLT" treatment. To determine why this treatment is necessary and successful, a series of two-step heat treatments was applied to 5.5Ni steel and the resulting microstructural states were characterized and compared with that obtained through the QLT treatment. It was concluded from this analysis that the QLT treatment lowers the ductile-brittle transition temperature by precipitating a dense distribution of thermally stable austenite along the boundaries of martensite laths, which interrupts the crystallographic alignment of laths within martensite packets and prevents cooperative trans-packet cleavage. Essentially, it reduces the mean free fracture path for cleavage. The multistep heat treatment is necessary because of the low nickel content; a single step heat treatment leads to an austenite precipitate which is either too lean in solute to be retained or too coarse in its distribution to be effective. The problem is avoided in the QLT treatment since the intercritical anneal (L) serves to create regions of high solute content along the prior martensite lath boundaries. The intercritical temper (T) then precipitates a dense distribution of high solute, stable austenite within these enriched regions.

I. INTRODUCTION

FERRITIC steels which are intended for structural use at cryogenic temperatures are thermally processed to lower the ductile-brittle transition temperature to below the intended temperature of service. In conventional 9 pct Ni steel this treatment involves a straightforward intercritical temper at a relatively low temperature within the two-phase (α + γ) region. During the intercritical temper a fine, dense distribution of austenite phase is precipitated along the boundaries of the dislocated laths of the martensite structure. This austenite is thermally stable on cooling to at least 77 K. It lowers the ductile-brittle transition temperature by breaking up the crystallographic alignment of the martensite laths within packets through a toughening mechanism described in Reference 1.

Ferritic steels of lower nickel content can also be toughened for cryogenic service as can ferritic iron-manganese steels containing no nickel at all. But in these cases more elaborate thermal treatments are required. The most commercially important of these alternative heat treatments is the "QLT" treatment. To determine why this treatment is necessary and successful, a series of heat treatments was performed using optical, transmission, and microscopic methods. The purpose of this work is to study the interplay of composition and microstructure in determining the ductile-brittle transition temperature of 5.5Ni steel, with particular emphasis on the benefit achieved from the QLT treatment.

II. EXPERIMENTAL PROCEDURE

The alloy used for this investigation was a commercial Fe-5.5Ni steel which was provided by the Nippon Steel Corporation. Its composition was determined to be: Fe-5.86Ni-1.21Mn-0.69Cr-0.20Mo-0.25Si-0.06C-0.02S-0.008P (in wt pct). The as-received alloy was annealed at 1200 °C for two hours to remove the effect of prior thermomechanical treatment. The alloy was then solution-annealed at 900 °C for two hours. Experimental samples of approximate dimension 10 cm × 10 cm × 3 cm were cut from the annealed plates and subjected to one of the four heat treatments diagrammed in Figure 1. Each of these heat treatments begins with a treatment labeled Q, which involves austenitization at 800 °C for one hour followed by rapid quenching in ice water. Subsequent intercritical tempering at 600 °C for two hours yields the treatment QT2. Intercritical tempering at 600 °C for 100 hours gives QT800. Intercritical annealing at 670 °C for one hour yields the treatment labeled QL. The QL treatment followed by an intercritical temper at 600 °C for one hour gives the treatment labeled QLT. In all cases the samples are quenched in ice water after heat treatment.

The microstructure of the alloys was studied as a function of heat treatment using optical, transmission, and
scanning transmission electron microscopy. The latter technique was used primarily to gain information about the chemical composition of the austenite present in the heat treated samples. The details and results of this analysis are reported elsewhere.6

The volume fraction of precipitated austenite was determined by X-ray diffraction using FeKα radiation. For quantitative analysis the (220) and (311) austenite peaks were compared with the (211) martensite peak by the technique suggested by Miller.7 The volume fraction of precipitated austenite was determined both in specimens quenched to room temperature and in specimens which have been soaked in liquid nitrogen. The austenite fraction was also determined by backscatter Mössbauer spectroscopy using the apparatus and techniques described by Fultz.8 The Mössbauer measurements were used to calibrate the X-ray determinations and to identify the residual austenite in the surfaces of broken fracture specimens.

The mechanical property measurements included the tensile properties and the impact toughness. Tensile tests were conducted at room temperature and at 77 K. The impact toughness was measured over a range of temperatures to determine the ductile-brittle transition temperature. The impact tests employed standard Charpy V-notch specimens prepared according to ASTM specifications.9 Fractographic analyses of the broken Charpy V-notch test specimens included scanning electron fractographic studies and measurements of the residual austenite content through backscatter Mössbauer spectroscopy.

### III. RESULTS

#### A. The Variation of Microstructure with Heat Treatment

When a sample of Fe-5.5Ni steel is heated into the two-phase (α + γ) region, it undergoes a partial retransformation to austenite. If the holding temperature is not too high, the austenite precipitation is accomplished by diffusional processes, with the consequence that the chemical composition of the precipitated austenite differs from that of the residual ferrite. On subsequent quenching a portion of this precipitated austenite may retransform martensitically. The final microstructure will then be a mixture of three constituents: tempered martensite, which represents that part of the alloy which did not retransform on heating; fresh martensite, which represents that part of the precipitated austenite which retransforms during cooling; and austenite, which represents that part of the precipitated austenite which is retained. The martensite and austenite phases can be distinguished from one another through crystallographic measurements, such as dark-field transmission electron microscopy, or magnetic measurements, such as Mössbauer spectroscopy. The fresh and tempered martensites can often be distinguished from one another through differences in microstructure. The separation is sometimes clear, but is never entirely unambiguous. The microstructural changes which result from heat treatment involve changes in the size, shape, distribution, and volume fractions of these three constituents and in their internal composition and structure.

1. **The Q Condition**

Figures 2 and 3 show the microstructure of the Fe-5.5Ni alloy in the reference or Q condition. Figure 2 is an optical micrograph. It reveals a lath martensite structure in which martensite laths are organized into aligned packets which subdivide the prior austenite grains. Figure 3 is a...