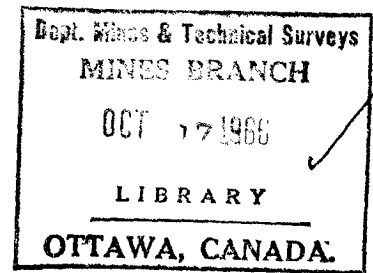




CANADA



**STUDY OF AS-ROLLED CARBON
STEELS OVER RANGES OF URANIUM
SULPHUR AND CARBON CONTENTS**

D. K. FAURSCHOU

**DEPARTMENT OF MINES AND
TECHNICAL SURVEYS, OTTAWA**

PHYSICAL METALLURGY DIVISION

MINES BRANCH

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STUDY OF AS-ROLLED CARBON STEELS OVER RANGES OF
URANIUM, SULPHUR AND CARBON CONTENTS

by

D. K. Faurschou*

ABSTRACT

Certain ad hoc studies of uranium in carbon steels at the Mines Branch have been complemented and clarified by statistically designed and analyzed studies. The results indicate that uranium has a very limited potential usefulness as an alloying element in ferrous metallurgy. Uranium has more potential as a scavenger and sulphide former.

A factorial set of as-rolled carbon steels having four uranium levels (nil, 0.15, 0.3 and 0.6%), three sulphur levels (0.006, 0.030 and 0.14%) and two carbon levels (0.14 and 0.34%) was tested to assess the influence of uranium on impact characteristics, active-state corrosion resistance, stress-corrosion resistance, machinability, microstructure, and isothermal transformation of austenite. The sulphur and carbon levels were introduced in anticipation of interaction effects with uranium as well as for hidden replication.

Uranium was always detrimental in progressively raising C_v fracture-appearance transition temperatures. However, only uranium contents exceeding 0.15% were inherently detrimental to absorption of impact energy. At the highest sulphur level, and particularly in transverse tests, uranium beneficially raised the curves of C_v impact energy versus temperature.

The rates of active corrosion in 5% HC 1 and H_2SO_4 were

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significantly but marginally reduced by uranium. Accelerated stress-corrosion results indicate some improvement related to the presence of 0.09% uranium.

Uranium in amounts sufficient to globularize sulphides did not significantly affect machinability. Uranium makes possible the production of ultra high sulphur steels having good transverse impact toughness.

In carbon steel, uranium refined the as-cast macrostructure, promoted the formation of degenerate pearlite and slightly delayed the transformation of austenite, but had almost no effect on tensile properties.

The factorial design of the impact test program permitted quantitative determination of the effects of sulphur and carbon on C_v 15 ft-lb and C_v 50% cleavage transition temperatures.

Direction des mines

Rapport de recherches R 178

ÉTUDE DES ACIERS AU CARBONE TELS QUE LAMINÉS,
SELON LEURS TENEURS EN URANIUM,
EN SOUFRE ET EN CARBONE

par

D. K. Faurschou*

RÉSUMÉ

Certaines recherches sur l'uranium dans les aciers au carbone, effectuées à la Direction des mines, ont été complétées et clarifiées au moyen d'études et d'analyses statistiques. Les résultats indiquent que l'uranium possède une utilité possible très restreinte comme élément d'addition dans la métallurgie de l'acier. Il offre de meilleures possibilités comme épurateur et générateur de sulfure.

Un jeu factoriel de pièces d'acier au carbone tel que laminé, comportant quatre teneurs en uranium (0, 0.15, 0.3 et 0.6 p. 100), trois teneurs en soufre (0.06, 0.30 et 0.14 p. 100) et deux en carbone (0.14 et 0.34 p. 100), a été mis à l'essai pour évaluer l'influence de l'uranium sur la résistance au choc, la résistance à la corrosion accélérée et à la corrosion sous contrainte, l'usinabilité, la microstructure, et la transformation isothermique de l'austénite. Les teneurs en soufre et en carbone ont été ajoutées dans l'attente qu'il se produirait des effets d'interaction avec l'uranium et des réactions cachées.

L'uranium s'est toujours montré nuisible en augmentant progressivement les températures de transition pour un clivage déterminé lors de l'essai sur l'éprouvette Charpy en V. Toutefois, seules les teneurs en uranium supérieures à 0.15 p. 100 sont en elles-mêmes nuisibles à l'absorption de l'énergie de rupture. Pour les pièces comportant la plus forte teneur en soufre et surtout lors des essais transversaux, l'uranium a élevé les courbes de l'énergie de rupture par rapport à la température lors de l'essai sur l'éprouvette Charpy en V.

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Les vitesses de corrosion accélérée dans l'HCl et l'H₂SO₄ à 5 p. 100 ont été réduites par l'uranium de façon significative mais peu prononcée. Les résultats de la corrosion accélérée sous contrainte indiquent une certaine amélioration par suite de la présence de 0.09 p. 100 d'uranium.

L'uranium en quantité suffisante pour globulariser les sulfures n'a pas d'influence notable sur l'usinabilité. L'uranium rend possible la production d'acier à très haute teneur en soufre qui présente une bonne résistance au choc lors de l'essai transversal.

Dans l'acier au carbone, l'uranium affine la macrostructure du brut de coulée, cause la formation de perlite dégénérée et retarde un peu la transformation de l'austénite mais ne possède à peu près aucun effet sur les propriétés de traction.

La nature factorielle du programme d'essais de choc a permis de faire la détermination quantitative des effets du soufre et du carbone sur les températures de transition, pour un travail de 15 pieds-livres et un clivage de 50 p. 100, lors de l'essai sur l'éprouvette Charpy en V.

CONTENTS

	<u>Page</u>
Abstract	i
Résumé	iii
Index to Tables and Figures	vi
Introduction	1
Experimental Steels	2
Procedures and Results	5
Macrostructure	5
Microstructure	5
Inclusions	6
Isothermal Transformation of Austenite	7
Active-State Corrosion	8
Specimens and Test Conditions	8
Results	8
Stress-Corrosion Cracking	13
Material and Test Conditions	13
Results	14
Machinability	15
Tensile Properties	16
Charpy Impact Properties	18
Transverse Impact Tests	18
Longitudinal Impact Tests	19
General Discussion	29
Conclusions	33
Acknowledgements	35
References	35
Figures 1-17	39-54

TABLES

<u>No.</u>	<u>Description</u>	<u>Page</u>
1.	Chemical Composition of Factorial Series of 24 Steels	4
2.	Raw Results of Active-State Corrosion Tests	9
3.	Summary of Analysis of Variance of Corrosion Tests in 5% HCl	10
4.	Summary of Analysis of Variance of Corrosion Tests in 10% H ₂ SO ₄	11
5.	Raw Tensile Results	17
6.	Raw Longitudinal Charpy V-Notch Impact Results for 12 Low Carbon Steels	21
7.	Raw Longitudinal Charpy V-Notch Impact Results for 12 Medium Carbon Steels	22
8.	C _v 15 ft-lb Transition Temperatures for 24 Steels	23
9.	C _v 50% Cleavage Transition Temperatures for 24 Steels	24
10.	Summary of Analysis of Variance of C _v 15 ft-lb Transition Temperatures	25
11.	Summary of Analysis of Variance of C _v 50% Cleavage Transition Temperatures	26
12.	Relevant 95% Confidence Limits and Least Significant Differences Applicable to the Experimental Transition Temperatures	27

FIGURES

<u>No.</u>	<u>Description</u>	<u>Page</u>
1.	Macrodistribution of Uranium-Rich Inclusion Galaxies (not sulphides) in 4-Inch Sand-Cast Ingots	39
2.	Effect of Uranium on the Etched Microstructure of As-Rolled Carbon Steel	40
3.	Effect of Uranium on Sulphide Inclusions in Wrought Carbon Steel	41
4.	TTT Curves of Low and Medium Carbon Steels, With and Without Uranium	42
5.	Effect of Uranium on TTT Curves of Low Carbon Steel	43
6.	Effect of Uranium on TTT Curves of Medium Carbon Steel	43
7.	Effect of Uranium on Resistance of Low and Medium Carbon Steels to Stress-Corrosion Failure in Boiling Aqueous Calcium Nitrate	44
8.	Effect of Uranium on Transverse Charpy V-Notch Impact Transition Curves of Low Carbon Steel	45
9.	Effect of Uranium on Transverse Charpy V-Notch Impact Transition Curves of Medium Carbon Steel	45
10.	Effect of Uranium on C_v Energy Absorption and Per Cent Cleavage Fracture at Low, Medium and High Sulphur Levels of Low Carbon Steels	46
11.	Effect of Uranium on C_v Energy Absorption and Per Cent Cleavage Fracture at Low, Medium and High Sulphur Levels of Medium Carbon Steel	47

Figures - continued -

<u>No.</u>	<u>Description</u>	<u>Page</u>
12.	Effect of Sulphur on C_V Energy Absorption at the nil, 0.14, 0.30 and 0.65% Uranium Levels	48 & 49
13.	Effect of Uranium on C_V 15 ft-lb and 50% Cleavage Transition Temperatures at the 0.006, 0.032 and 0.139% Sulphur Levels	50
14.	Effect of Uranium on the C_V 15 ft-lb and 50% Cleavage Transition Temperatures at Low and Medium Carbon Levels	51
15.	Effect of Sulphur on C_V 15 ft-lb and 50% Cleavage Transition Temperatures at the nil, 0.14, 0.30 and 0.65% Uranium Levels	52
16.	Effect of Carbon on C_V 15 ft-lb and 50% Cleavage Transition Temperatures at the nil, 0.14, 0.30 and 0.65% Uranium Levels	53
17.	Effect of Carbon and Sulphur on the C_V 50% Cleavage Transition Temperature at the 0.006, 0.032 and 0.139% Sulphur Levels and the 0.14 and 0.37% Carbon Levels, respectively ..	54

INTRODUCTION

Studies of the non-nuclear uses of natural uranium commenced in 1959 at the Mines Branch. These studies were undertaken primarily because of economic and political pressures occasioned by collapse of the major market for Canadian uranium. The facts that it was anticipated that large new markets would be developed by about 1970 and that large amounts of relatively inexpensive depleted uranium were available from the U.S.A. and the U.K. meant that the research effort would solve the immediate problem only if a large-volume use could be discovered within a short time.

As a consequence, efforts at the Mines Branch were concentrated first on the possibility of using uranium in carbon steel. Only carbon steel promised a large enough market. Surveys of the literature and patents (1, 2) offered some hope for success and some basis for organized experimentation. Due to the importance of quickly evaluating the influences of uranium and due to the lack of fundamental information about relevant uranium compounds and the physical chemistry of uranium in steel, much of the previously reported work was of an ad hoc nature⁽³⁾.

The ad hoc studies, of course, were designed to discover large effects related to small additions of uranium. After all, uranium is expensive. The studies were therefore inherently under-designed to uncover marginal effects and cope with interference from macrosegregation. Hence, some of the results were anomalous. Also, it appears that fate capriciously teased the investigators with initially promising results.

The ad hoc studies quickly showed that the interim marketing problems of the uranium industry could not be solved within the steel industry. Nevertheless, the momentum of the studies dictated their continuance on a reduced scale.

The present report concerns a follow-up of some of the initial studies. The work was intended to clarify some of these results and to evaluate more fully the marginal effects of uranium on the chance that uranium might have a unique if minor usefulness in carbon steel.

The factorial design of the work did, largely as a bonus, allow some purposeful evaluation of the influences of sulphur and carbon as well as uranium. The variables of sulphur and carbon were included because there was an expectation that the effects of uranium would be dependent on the sulphur and carbon contents; and because the effects of sulphur and of carbon were of direct interest. The inclusion of sulphur

and carbon as controlled variables did not significantly enlarge the size of the experiment, due to the fact that replication was necessary, in any event, to evaluate the marginal effects of uranium quantitatively. The information relating to the effects of sulphur and carbon was therefore largely evaluated as a consequence of so-called "hidden" replication.

The work involved examination of the as-cast and wrought macrostructures and microstructures; determination of critical allotropic transformation temperatures and isothermal transformation curves; and evaluation of active-state corrosion resistance, resistance to stress-corrosion, machinability, and impact toughness.

Conscious efforts were made to avoid duplication of the efforts of others and to make the factorial set of steels available to colleagues. While the present work was in progress, colleagues were continuing their investigations of the influences of uranium in carbon steel^(4, 5, 6, 7, 8, 9, 10, 11).

EXPERIMENTAL STEELS

Each heat was made individually in a 50-lb induction furnace and cast directly into a hot-topped dry-sand mould. The ingots were 4 inches in diameter by 9 inches high. The mould was developed for this project to achieve a solidification rate slow enough to permit significant macro-segregation^(12, 13, 14 and 15) of uranium-rich inclusions (except sulphides) to the extreme lower region of the ingot and so allow the use of relatively clean cropped ingots of a size that could be forged and rolled at the Mines Branch.

Figure 1 shows four autoradiographs which illustrate the effectiveness of this technique. These are typical autoradiographs of the central longitudinal plane. At 0.15% uranium the radioactive inclusions are well dispersed. At 0.34, 0.53 and 0.76% uranium most of the radioactive inclusions reside in the lower 10, 10 and 20% of each respective ingot.

A factorial set of 24 individual heats was produced to include all combinations of 4 levels of uranium, 3 levels of sulphur and 2 levels of carbon. The nominal levels of each factor were predetermined. The steels were produced randomly, as far as possible, in random groups of 4 or 5. The average uranium levels were nil, 0.14, 0.30 and 0.65%. The average sulphur levels were 0.006, 0.032 and 0.139%. The average carbon levels were 0.14 and 0.37%. This factorial design was chosen to

account for suspected first-order interactions. The design is efficient because it allows the effects of uranium to be determined by hidden replication and incidental information is made available concerning sulphur and carbon. The second and third-order interactions provide an estimate of the random experimental error.

All of the ingots had nominal contents of 0.65% manganese, 0.25% silicon and 0.006% phosphorus. The steels were deoxidized with 0.1% aluminum. The uranium recoveries were approximately 65, 70 and 75% at the 0.15, 0.30 and 0.65% uranium levels, respectively.

The results of chemical analyses of the basic 24 heats are given in Table 1.

The ingots were forged to 3-1/2 in. x 2 in. bars and hot-rolled to 1/2 in. plate. The two ingots having nominal levels of 0.005% sulphur, and the two ingots having 0.03% sulphur in combination with 0.6% uranium, had to be forged and rolled from below 1060°C (1950°F) because of the presence of too much intergranular UFe_2 (although UFe_2 melts about 1200°C (2200°F) it forms an Fe- UFe_2 eutectic, which melts at 1077°C (1976°F)). The other steels were forged from 1230°C (2250°F) and hot-rolled from 1150°C (2100°F). Efforts were made, using an optical pyrometer, to terminate rolling in the range of 870-900°C (1600-1650°F). The steels were tested in the as-rolled condition.

TABLE 1

Chemical Composition of the Factorial Series of 24 Heats

Heat	Element, Weight Per Cent									
	C	S	U	U/S Ratio	Mn	Si	P	Al	Ni*	Cu*
A	0.14	0.008	-	-	0.65	0.18	0.002	0.08	0.04	0.005
B	0.15	0.008	0.12	15	0.68	0.26	0.003	n.a.	n.a.	n.a.
C	0.15	0.004	0.25	62	0.67	0.17	0.003	0.10	0.04	0.006
D	0.14	0.005	0.50	100	0.72	0.25	0.004	0.10	0.05	0.006
E	0.13	0.029	-	-	0.56	0.07	0.008	0.08	0.04	0.12
F	0.14	0.031	0.14	4.5	0.61	0.22	0.002	n.a.	n.a.	n.a.
G	0.12	0.030	0.25	8.3	0.60	0.19	0.007	0.09	0.08	0.11
H	0.12	0.041	0.55	13.4	0.73	0.35	0.006	0.10	0.09	0.12
I	0.14	0.128	-	-	0.64	0.16	0.010	n.a.	n.a.	n.a.
J	0.12	0.116	0.13	1.1	0.59	0.12	0.009	n.a.	n.a.	n.a.
K	0.11	0.147	0.28	1.9	0.64	0.12	0.008	n.a.	n.a.	n.a.
L	0.16	0.142	0.67	4.7	0.64	0.19	0.011	n.a.	n.a.	n.a.
M	0.38	0.006	-	-	0.65	0.21	0.002	0.09	0.04	0.01
N	0.36	0.006	0.15	25	0.60	0.29	0.006	0.10	0.10	0.02
O	0.38	0.009	0.37	41	0.61	0.28	0.006	0.11	0.04	0.01
P	0.39	0.005	0.75	150	0.59	0.40	0.004	0.10	0.04	0.01
Q	0.38	0.031	-	-	0.72	0.21	0.003	n.a.	n.a.	n.a.
R	0.39	0.028	0.17	6.1	0.62	0.14	0.011	n.a.	n.a.	n.a.
S	0.37	0.027	0.32	12	0.64	0.19	0.012	n.a.	n.a.	n.a.
T	0.39	0.043	0.72	16.8	0.76	0.42	0.008	n.a.	n.a.	n.a.
U	0.34	0.142	-	-	0.71	0.24	0.009	n.a.	0.07	0.12
V	0.37	0.147	0.15	1.0	0.70	0.31	0.008	n.a.	n.a.	n.a.
W	0.35	0.150	0.36	2.4	0.71	0.24	0.003	n.a.	n.a.	n.a.
X	0.33	0.142	0.68	4.8	0.65	0.21	0.011	0.11	0.06	0.11

n.a. - not analyzed chemically.

*Heats A, B, C, D and M, N, O, P were made using electrolytic iron. All other heats were made using a special supply of Stelco billets.

PROCEDURES AND RESULTS

Macrostructure

Judging from the appearance of cross-sectional discs cut from the as-cast billets and etched in 1:1 HCl and water for 30 minutes at 70-80°C (160-180°F), one would conclude that uranium has a beneficial effect on as-cast structure which would be related to improved forgeability. In both the low and medium carbon steels the presence of 0.3 or 0.6% uranium appeared to markedly refine austenitic grain size and almost eliminate dendritic segregation. In the high sulphur steels the effect of uranium in refining grain size and obscuring dendritic structure was not as pronounced as in the low and medium sulphur steels.

Conventional sulphur prints show that uranium does not cause macrosegregation of sulphides. Steel having 0.15% sulphur plus 0.6% uranium produces a uniformly very-faint brown sulphur print. The print is faint because uranium makes the sulphides almost resistant to dilute sulphuric acid.

Autoradiography of cross-sectional discs cut from just below the riser showed a relatively uniform radial distribution of uranium, except in the steels having high uranium combined with low and medium sulphur. In these four steels the central-third region was very noticeably high in uranium. (These were the steels which had to be remade and hot-worked below 1060°C or 1950°F.)

Microstructure

The only readily observed effects of uranium on the etched microstructure of the as-rolled steels were tendencies to promote the formation of degenerate pearlite and veining substructure in the ferrite. These effects are readily noticeable with 0.3 and 0.6% uranium in low sulphur steel. They were not observed in high sulphur steel.

Figure 2, on the left-hand side, shows the normal lamellar pearlite observed in the as-rolled steels without uranium. By contrast, the right-hand side shows the comparable structure modified by 0.37% uranium. The uranium causes the pearlite to occur in angular patches and inhibits most of the carbide lamellae from developing into regular plates. The degenerate pearlite produced by uranium appears to be similar to the degenerate pearlite produced under certain circumstances by molybdenum⁽¹⁶⁾. The pearlite of both micros is over etched. However, to properly reveal veining substructure requires even heavier etching. Some substructure is just emergent in the ferrite grains of the uranium steel. Such subgrain boundaries in various metals have been recognized

to originate as dislocation walls separating blocks of slightly different crystallographic orientation.

The ASTM ferrite grain size of the as-rolled steels, despite the appearance of the deep-etched sections, was unaffected by uranium. In the low carbon steels the grain-size range was 6-1/2 to 7-1/2. In the medium carbon steels the grain-size range was 7 to 8.

Inclusions

It was intended to study uranium-bearing inclusions, particularly sulphides, in detail. It was reasoned that because there were indications that uranium might be useful at low levels, below 0.10%, the recovery of uranium might be very dependent on the sulphur, oxygen and nitrogen content of the steel. It was thought, for example, that consistent recovery of uranium to inhibit stress-corrosion failure or to scavenge oxygen and nitrogen would depend upon the sulphur level. However, extraction of uranium compounds necessitates the use of non-aqueous media and inert atmospheres. C.E. Makepeace of Eldorado was at the time actively developing the required apparatus and techniques⁽¹⁷⁾. Also, intensive study of inclusions, if warranted, was deferred in anticipation of the availability of electron probe and electronic phase scanning equipment. Accordingly, the following observations of inclusions were essentially qualitative.

Heavy alumina stringers were observed in all of the steels without uranium. The content of alumina inclusions, particularly at low and medium sulphur levels, was very considerably reduced by 0.15% uranium.

The effect of uranium is most profound on sulphides. In all of the as-rolled steels at the nil uranium level the sulphides were elongated and grouped in stringers. In the low sulphur steels, 0.15% uranium eliminated sulphide stringers. In the medium sulphur steels, with 0.15% uranium, the sulphides were a mixture of elongated, semi-elongated and globular forms. At 0.3% uranium the sulphides were completely globular in the medium sulphur steels. In the high sulphur steels, with 0.15% uranium the sulphides were of the elongated and semi-elongated forms, with 0.3% uranium the sulphides were of the semi-elongated and globular forms, and with 0.6% uranium the sulphides were globular.

Figure 3 illustrates the effect of uranium on alumina content and on sulphide inclusions at the high sulphur level. The steel having 0.13% uranium is much cleaner than the steel without uranium. Also, the morphology of the sulphides changes from stringers of elongated simple sulphides to an almost random distribution of globular, complex sulphides. These changes are progressive as the uranium content increases.

In all uranium-bearing steels, particularly at low sulphur levels and 0.3% or more uranium, stringers of U (OCN) complex inclusions may be observed. These inclusion stringers are similar in size and occurrence to alumina particles and stringers.

The occurrence and amount of observable UFe_2 depend upon the uranium and sulphur contents. (There is evidence, from quench-aging tests done by G.P. Contractor^(10, 11), that some UFe_2 exists in submicroscopic form.) In the low sulphur steels, UFe_2 was observed at the 0.3 and 0.6% uranium levels. In the medium sulphur steels UFe_2 was only observed at the 0.6% uranium level. No UFe_2 was observed in the high sulphur steels. Also, no UC was observed, unless a pale coppery-coloured cubic type of inclusion, often associated with UFe_2 , is UC.

In the steels which could only be forged below $1065^{\circ}C$ ($1950^{\circ}F$), the UFe_2 was sometimes present as "easter eggs" of Fe- UFe_2 eutectic. UFe_2 has a pale grey colour, which makes it hard to observe in unetched specimens or even in normally etched specimens. However, if the specimen is etched at 6 volts in alkaline sodium picrate, every phase except UFe_2 will acquire a brown stain or be attacked. The cubic phase suspected of being UC will be heavily attacked by this etching technique.

Isothermal Transformation of Austenite

Isothermal transformation curves were determined by M. J. Walker⁽¹⁸⁾ for a pair of the low and a pair of the medium carbon steels. One steel of each pair was free of uranium, while the other contained a nominal of 0.3% uranium. The pairs of steel were selected for their matching chemical composition. For each pair the calculated hardenability, excluding a factor for uranium, of the steel with uranium was slightly greater than that of its mate without uranium. The difference of 0.04 in multiplying factors is equivalent to the effect of only about 0.10% Ni or 0.06% Si or 0.02% Cr or 0.015% Mo. The specimens were 30 mils thick. They were coated electrolytically with one mil of copper to inhibit carburization or decarburization of the surfaces during heat treatment.

In Figure 4⁽¹⁸⁾, TTT curves for the low-carbon medium-sulphur steels are paired at the top, while those for the medium-carbon low-sulphur steels are paired at the bottom. The curves for the uranium-bearing steels are at the right. At both carbon levels the uranium appears to have caused a modest shift, to longer times, of the complete diagram without significantly altering its shape. The modest stabilization of the austenite is understandable because uranium is known to have low solubility even in austenite. The U-Fe phase diagram shows the solubility of uranium in gamma decreasing from almost 2% at $1370^{\circ}C$ ($2500^{\circ}F$) to virtually zero at $955^{\circ}C$ ($1750^{\circ}F$).

Figures 5 and 6, based on Walker's data, allow more direct comparison of the TTT curves of the paired steels. Figure 5 shows that the effect of 0.25% uranium was very small. Figure 6 shows that the medium-carbon steel with 0.37% uranium started to transform when the steel without uranium was transformed about 25%, and the steel with 0.37% uranium was about 25% transformed when the steel without uranium was 99% transformed.

Active-State Corrosion

Previously, results of corrosion tests of uranium-bearing steel tended to produce erratic or even anomalous results^(3, 19, 20, 21), with respect to corrosion rate and sensitivity to pitting. A principal reason was that sufficiently-well controlled or matched and oriented sets of specimens were not available. Also, the results, especially the unfavourable ones, were viewed with suspicion because of the extent of macro-segregation in the conventional small ingots.

Specimens and Test Conditions

A 12 in. x 4 in. length from the mid-length of each 1/2 in. plate was hot-rolled to 1/8 in. Ten corrosion specimens, each 2 in. x 1-3/4 in. x 0.080 in., were prepared from each steel. The long dimension always lay in the direction of rolling. The surfaces were ground to a uniformly fine finish. Autoradiography was used to record the distribution of uranium and to check the identity of the groups of specimens. The specimens were cleaned, weighed, measured and degreased just prior to testing.

The specimens were randomly arranged in separate but complete sets of 24 for each test. The tests consisted of simple immersion in 5% HCl for 12, 24, 48 and 96 hours; in 10% H₂SO₄ for 3, 6 and 12 hours; and in 10% HNO₃ for 1, 2 and 4 hours. At intervals the acid was replenished to maintain its concentration within an estimated 15% of the nominal concentration. Control of the tests was insufficient to study how corrosion rate varied with time, as had been planned.

The value of testing carbon steel in such active media as 5% HCl and 10% H₂SO₄ is subject to justified criticism. In this work it was done to settle controversy over previous screening results here^(3, 19) and in Japan^(20, 21).

Results

The individual test results, calculated in inches of metal loss per year (ipy), are recorded in Table 2. The results of analysis of variance of average corrosion rates in 5% HCl and 10% H₂SO₄ are presented in Tables 3 and 4, respectively.

TABLE 2

Individual Results of Active-State Corrosion Tests

Steel	Corrosion Rates, ipy										
	5% HCl				10% H ₂ SO ₄				10% HNO ₃		
	12 hr	24 hr	48 hr	96 hr	3 hr	6 hr	12 hr	24 hr	1 hr 3-3/4 min	2 hr 7-1/2 min	4 hr 15 min
A	0.4683	0.4481	0.4477	0.5320	1.607	1.304	1.469	1.491	66.31	55.88	34.15
B	0.4996	0.4508	0.4420	0.4943	1.448	1.193	1.371	1.408	69.48	50.00	30.22
C	0.4191	0.3967	0.3967	0.4587	1.508	1.245	1.414	1.445	71.42	53.35	29.54
D	0.4086	0.4130	0.3901	0.4415	1.547	1.293	1.447	1.434	69.18	59.28	42.59
E	0.1300	0.1371	0.1516	0.2219	0.2827	0.1599	0.1568	0.1494	59.92	46.89	35.11
F	0.1187	0.1445	0.1524	0.2096	0.2069	0.1467	0.1366	0.1285	68.52	56.54	31.64
G	0.1090	0.1155	0.1397	0.1867	0.1911	0.1314	0.1181	0.1125	69.71	60.29	38.69
H	0.1322	0.1423	0.1700	0.2280	0.4182	0.2636	0.2447	0.2575	60.05	65.64	31.24
I	0.1810	0.1902	0.2680	0.3646	0.4393	0.2803	0.3119	0.2935	55.83	39.86	36.13
J	0.1441	0.1437	0.1828	0.2324	0.3427	0.2210	0.1933	0.1986	69.00	55.05	36.08
K	0.1236	0.1274	0.1494	0.2276	0.3023	0.1994	0.1731	0.1586	55.68	45.72	30.94
L	0.1441	0.1516	0.1850	0.2504	0.3642	0.2860	0.2689	0.2465	74.66	49.41	30.83
M	0.5046	0.4226	0.3985	0.4020	1.362	1.134	1.314	1.365	65.52	51.03	28.34
N	0.3291	0.3664	0.3370	0.3234	1.035	0.8747	1.057	1.094	62.79	55.86	33.05
O	0.4367	0.3831	0.3396	0.3260	1.259	1.027	1.228	1.224	69.87	57.87	41.38
P	0.5276	0.4472	0.3853	0.3880	1.442	1.196	1.441	1.373	72.22	51.86	37.92
Q	0.1885	0.2104	0.2794	0.3225	0.2930	0.2126	0.2214	0.2078	65.77	46.42	33.27
R	0.1472	0.1511	0.1819	0.2034	0.2913	0.1744	0.1907	0.1687	75.68	53.91	37.52
S	0.1494	0.1432	0.1709	0.1968	0.3036	0.1973	0.1907	0.1731	75.45	57.81	38.96
T	0.2157	0.2113	0.2509	0.2829	0.4481	0.3361	0.3282	0.3269	63.89	58.00	41.11
U	0.2640	0.3365	0.5232	0.6572	0.5070	0.3545	0.4508	0.5184	47.95	42.64	29.15
V	0.2249	0.2574	0.3844	0.4920	0.4262	0.2834	0.3088	0.3559	55.85	49.17	35.79
W	0.2183	0.2491	0.3638	0.4472	0.4358	0.2711	0.3317	0.3827	73.21	46.77	29.67
X	0.2030	0.2271	0.2759	0.3712	0.4850	0.2987	0.3607	0.3550	77.18	50.81	32.11

TABLE 3

Summary of Analysis of Variance of the Average Corrosion Rates
(average ipy for 12, 24, 48 and 96 hr tests) of Steels A to X in 5% HCl

Source of Variation	d.f.	Sums of Squares	Mean Squares	F Ratio	Significance Level, α	100 (1 - α)*, %
Uranium	3	0.2985376	0.0995123	4.95	0.025	97.5
Sulphur	2	3.7297350	1.8986750	94	0.005	99.5
Carbon	1	0.2511874	0.2511874	12.5	0.005	99.5
U x S	6	0.1314168	0.02109028	1.04	n. s.	
U x C	3	0.0544205	0.01814016	0.86	n. s.	
S x C	2	0.6477039	0.32385195	15.4	0.005	99.5
U x S x C	6	0.1258105	0.020968416			
Total	23	5.2388117				
Pooled Residual (U x S x C, U x C and U x S)	15	0.3116478	0.0207765			

*100 (1 - α) is the percentage confidence level or probability that a true effect or result has been detected.

n. s. - not statistically significant.

TABLE 4

Summary of Analysis of Variance of the Average Corrosion Rates
(average ipy for 3, 6, 12 and 24 hr tests) of Steels A to X in 10% H₂SO₄

Source of Variation	d. f.	Sums of Squares	Mean Squares	F Ratio	Significance Level, α	100 (1 - α)*, %
Uranium	3	1.1477236	0.3825745	7.0	0.005	99.5
Sulphur	2	92.8559597	46.4279798	982	0.005	99.5
Carbon	1	0.0064321	0.0064321	0.0012	n. s.	
U x S	6	0.4928695	0.0821449	1.74	0.25	75
U x C	3	0.0446936	0.0148979	0.31	n. s.	
S x C	2	1.8132512	0.9066256	19.2	0.005	99.5
U x S x C	6	0.2835781	0.0472630			
Total	23	96.6445078				
Pooled Residual (U x S x C, U x C and U x S)	15	0.8211412	0.0547427			

* 100 (1 - α) is the percentage confidence level or probability that a true effect or result has been detected.

n. s. - not statistically significant.

Analysis of variance of the individual results obtained in HCl and H₂SO₄ revealed that the random residual error for the tests was very small. Even the pooled non-significant residual values in Tables 3 and 4 are very small. This proved that effects due to segregation of uranium were inconsequential in these tests. The tests were sensitive enough to show that the effect of uranium was highly significant in a statistical sense; although, in a practical sense, it was evident that the beneficial effect of uranium was marginal.

Tables 3 and 4 reveal that the principal source of variation in the corrosion rates, to the extent of 71% in the 5% HCl and 96% in the 10% H₂SO₄, was the sulphur content. However, this is misleading as the mean square values are composite quantities and particularly as the effect of sulphur was confounded with the effect of different melting stocks.

The most interesting result was apparently related to the melting stocks rather than to uranium or to sulphur. The low sulphur steels (A, B, C, D and M, N, O, P) were made from electrolytic iron. The other steels were made from one order of Stelco billets. It is unlikely that low sulphur content would greatly affect general corrosion rates in these tests. Yet, the corrosion rates of the steels made from electrolytic iron were about three to six times greater than for steels made from Stelco billets. The reason for this behaviour is probably the difference in copper levels. Chemical and spectrographic analyses showed that the only significant difference in the contents of residual elements occurred in the copper contents. The steels made from electrolytic iron analyzed from 0.005 to 0.02% copper, while the steels made from Stelco billets analyzed 0.11 to 0.12% copper. E. Williams and M.E. Komp⁽²²⁾ report that in 42% H₂SO₄ the corrosion rate of carbon steel containing 0.02% copper is up to sixteen times greater than that of carbon steel containing 0.10% copper. Their average corrosion rates were similar (0.15 to 3.0 ipy) to those reported herein for 10% H₂SO₄.

In 5% HCl the overall mean corrosion rate was 0.59 ipy. Uranium reduced this mean corrosion rate by 9, 20 and 14% at the 0.15, 0.3 and 0.6% uranium levels, respectively. At 0.36% carbon the corrosion rate was 20% greater than at 0.14% carbon. At low-sulphur (electrolytic iron), medium-sulphur and high-sulphur levels the respective rates of corrosion were in the ratio of 2.85 to 1.00 to 1.23. The S x C interaction was highly significant and this is interpreted as indicating that the sulphur caused the medium carbon steel to have a higher mean corrosion rate than the low carbon steel. The U x S interaction was non-significant.

In 10% H₂SO₄ the overall mean corrosion rate was 2.48 ipy. This mean corrosion rate was reduced 16% by 0.15% uranium and reduced 12% by 0.3% uranium. The corrosion rate was increased 4% by 0.6% uranium.

There was a suggestion of a U x S interaction, which made sulphur less detrimental in the presence of uranium (the level of significance was only 0.25). At the low-sulphur (electrolytic iron), medium-sulphur and high-sulphur levels the respective rates of corrosion were in the ratios of 5.9 to 1.00 to 1.45. The mean corrosion rates were not affected by carbon.

In 10% HNO₃ the corrosion rates were so extremely high, averaging 1390 ipy, and the apparent differences were so slight, that the results were not analyzed statistically.

Visual differences were observed in the extent of pitting in 5% HCl. These differences were slight on the as-rolled surfaces. However, on the end-grain surfaces pitting became increasingly severe as the sulphur content increased, unless the uranium content was also increased. At the high-sulphur levels the end-grain attack was severe at the nil uranium level; however, at the high-sulphur plus high uranium levels the end-grain surfaces were almost free of pitting attack.

Stress-Corrosion Cracking

W. A. Morgan, R. D. McDonald and G. P. Contractor had applied for patents⁽²³⁾ involving the use of uranium to reduce the susceptibility of steel to failure by stress corrosion; but, as the results of stress-corrosion tests are not always easy to interpret^(3, 4), it seemed expedient to investigate the behaviour of the factorial set of carbon steels.

Material and Test Conditions

The existing 24 heats were supplemented by 6 heats representing the 2 levels of carbon and 3 levels of sulphur at a nominal level of 0.08% uranium. All 30 heats were tested.

As with the corrosion tests, specimens were prepared from hot-rolled 1/8 in. plate. The specimens, 3-1/2 in. x 3/16 in. x 40 mils, were bent 173 degrees over a 1/2 in. diameter mandrel and inserted into a Teflon retaining holder which was grooved to accommodate the ends of the specimens. Care was taken to bend the specimens directly to fit the holder, to avoid disturbing the as-bent stress distribution. The specimens were tested in groups of one to four complete sets. Ten complete sets were tested.

The test medium was a boiling aqueous solution of 60% Ca (NO₃)₂ - 3% NH₄OH. This medium was chosen because experience of I. G. Farbenindustrie had shown that if a steel resisted this test for more than 200 hours it was immune to stress-corrosion cracking in common industrial applications.

Results

The life of individual specimens was extremely variable. The sets of specimens were examined at regular intervals to take note of the incidence of failure and to remove failed specimens. Assessment of the results was complicated by the fact that the early failure rates were higher than anticipated. Inspection should have commenced earlier and been more frequent. However, plotting the results as cumulative per cent failure on logarithmic normal graph paper appears to offer a satisfactory comparison of the results at each of the uranium levels as shown in Figure 7.

The log of time to failure is plotted along the horizontal axis. The cumulative per cent frequency of failure is plotted up the vertical axis. When data plotted in this way fall on a straight line it means that the data form a Normal, i. e., Gaussian distribution. Such a distribution is illustrated by the bell-shaped curve along the left side of Figure 7. The scale on the right side shows the $\pm 1, 2$ and 3 sigma limits of a Normal distribution.

Figure 7 shows that the group of steels containing 0.09% uranium are markedly superior to any of the other groups. For example, 50% of the steels containing 0.09% uranium survive beyond 40 hours, but 50% of the steels without uranium survive only 4.4 hours. Hence, at the 50% survival point the steel with 0.09% uranium has a 9-fold advantage over the steel without uranium. This advantage decreases when comparisons are made at longer times, because the curves, if extrapolated, appear to converge toward 600 hours.

Figure 7 also shows that the groups of steels having 0.14, 0.3 and 0.6% uranium are inferior to the group of steels without uranium. The extent of this inferiority increases as these uranium contents increase.

All of the curves are disturbing when one questions their practical significance. For one thing, the life expectancy of individual specimens in any group is very wide, ranging from a few minutes to over a hundred hours. There does not appear to be a threshold value below which failure will not occur. On this account the practical superiority of the 0.09% uranium steel may be questioned, in the absence of actual service data. It may be of relevance to note that all of these steels were also tested after being cold-rolled 60% and every specimen survived the critical 200-hour exposure.

Machinability

Contractor, King and McClure⁽²⁴⁾ patented the use of uranium to improve the transverse mechanical properties of a broad range of steels.

It was considered that uranium, by controlling sulphide morphology, might significantly improve the machinability of steel. It is commonly, but erroneously, believed that stringers of highly elongated sulphides necessarily act as chip breakers and thereby improve machinability. However, it has been shown by Van Vlack⁽²⁵⁾ and others, that globular-type sulphides are more effective in carbon steels than more highly elongated (more plastic) sulphides. Merchant and Zlatin⁽²⁶⁾ have shown that sulphides improve machinability by reducing the coefficient of friction between tool and work. The reduced coefficient of friction reduces the energy absorption, which in turn causes the chips to curl tighter and break more frequently due to mechanical interference. Uranium not only causes the formation of globular sulphides but also reduces the tendency to formation of sulphide stringers. In fact, the globular uranium-bearing sulphides are predisposed to a random distribution in the steel.

Accordingly, when it became possible to incorporate some machinability logs into another program four additional heats were made. These heats were replicates of heats I, L, U and X in the main series. The tests thus compared the effect of 0.6% uranium versus no uranium in high sulphur steels having a low and a medium carbon level.

Each 4 in. diameter ingot was forged to 2-1/4 in. in diameter and rough machined to 2 in. by 20 in. SAE machinability ratings were determined at Battelle, using a constant-pressure lathe and B1112 steel as a reference standard.

This comparative machinability rating failed to show any influence due to uranium. It would appear that the effect of favourable sulphide morphology was offset by unfavourable effects also related to uranium. It is possible, for example, that the complex globular sulphides are not good lubricants, as are simple manganese sulphides.

It is recognized⁽²⁷⁾ that the major usefulness of tool-force data has been in the development of data necessary for the design and selection of machine tools and their accessories. Tool-force data may or may not relate in a meaningful way to production-run machinability ratings, depending on the operation and the grade of steel. A principal explanation for this is that the tool-force test does not consider surface finish, yet surface finish governs the permissible production rate. Considering this factor it might be expected that globular sulphides, as produced by uranium,

might permit considerably higher production rates than stringers of elongated sulphides would permit for a given surface finish.

Reference 27 also presents graphs which show an almost linear improvement in production rate, from 225 parts per hour with 0.15% sulphur to 350 parts per hour with 0.28% sulphur, in an AISI B1100 series steel. This suggests that uranium might be beneficially used to produce steels having ultra high sulphur levels which would have exceptionally good machinability. The uranium would serve to make these ultra high sulphur steels commercially forgeable as well as to control the morphology of the sulphides.

Tensile Properties

The longitudinal tensile properties of the factorial series of steel are given in Table 5. The bars were machined from as-rolled half-inch plate. Each reported result is the average of three tests. These results indicate the satisfactory quality and uniformity of the experimental steels.

It has been reported that uranium has no effect on tensile properties(3, 6, 19, 20). This has been confirmed at the 0.15 and 0.3% uranium level. However, at the high 0.6% uranium level the ratio of yield point to ultimate tensile strength was decreased by an overall average of 8%. This decrease, due entirely to uranium, was statistically significant at the 5% level. Such a decrease is qualitatively consistent with the effect of uranium on the morphology of pearlite.

TABLE 5

Average Longitudinal Tensile Properties of Steels A to X

Steel	% C	% S	% U	UTS (kpsi)	YP (kpsi)	YP UTS	% El in 2 in.	RA, %
A	low	low	none	62.7	46.7	0.745	34.7	72.3
B	"	"	0.12	64.9	45.7	.705	34.7	69.5
C	"	"	0.25	64.3	47.2	.735	31.2	74.8
D	"	"	0.50	65.3	43.7	.670*	31.2	73.9
E	"	med.	none	57.4	43.2	.753	38.2	70.8
F	"	"	0.14	60.1	44.2	.736	34.0	67.9
G	"	"	0.25	59.3	40.4	.682*	36.5	70.7
H	"	"	0.55	65.5	43.2	.660*	29.9	71.1
I	"	high	none	59.1	40.7	.688	32.8	65.2
J	"	"	0.13	54.4	38.7	.711	35.0	69.0
K	"	"	0.28	59.9	37.6	.698	34.5	70.7
L	"	"	0.67	60.8	41.1	.675*	33.5	65.0
M	med.	low	none	86.2	52.4	.607	25.8	57.3
N	"	"	0.15	83.5	50.4	.604	25.2	54.4
O	"	"	0.37	84.4	50.9	.604	26.3	58.5
P	"	"	0.75	85.1	49.7	.585*	26.0	54.9
Q	"	med.	none	84.6	52.8	.624	25.7	54.4
R	"	"	0.17	83.1	49.1	.591	26.7	55.7
S	"	"	0.32	83.6	47.2	.565*	25.8	56.2
T	"	"	0.72	90.1	51.7	.575*	23.0	52.5
U	"	high	none	79.8	48.9	.613	26.0	50.7
V	"	"	0.15	80.1	50.0	.623	26.2	51.1
W	"	"	0.36	81.3	49.2	.605	24.7	51.8
X	"	"	0.68	79.3	44.2	.547*	26.5	53.3

* Low ratios of $\frac{YP}{UTS}$ associated with the 0.3 or the 0.6% uranium level or both.

Charpy Impact Properties

In general, previously reported data^(3, 9) left some doubt as to the influence of uranium on impact characteristics. This was due to the inherent lack of discrimination of the ad hoc tests and to the generally wide variability of impact data in the transitional temperature range between tough and brittle fracture regions.

The results of transverse and of longitudinal tests are presented separately because evaluation of the longitudinal impact properties required statistical design and analysis, whereas evaluation of the transverse impact properties could be done by ad hoc experiments. Ad hoc tests were applicable to the transverse impact properties because the effects due to changes in the level of the uranium content were so large that they were readily detected without recourse to statistics. Also, the transverse tests were simpler to interpret because they were only concerned with the effect of uranium.

The results of the longitudinal tests are then presented so as to illustrate many of the advantages of a statistical approach over a classical approach in designing and analyzing highly variable and overlapping multi-variate data.

Transverse Impact Tests

The high sulphur steels having low and medium carbon contents (I, J, K, L and M, N, O, P) were subjected to transverse Charpy V-notch impact testing before the results of machinability tests on replicate steels were known. It was considered that if uranium improved machinability it would be of interest to know how uranium affected transverse properties. It was anticipated that resulphurized steel having globular-type sulphides would have impact properties superior to those of conventional resulphurized steels having stringers of thin elongated sulphides.

The test bars were machined transversely from 3/4-inch plate. The notches were normal to the plane of rolling. Three impact bars were broken at each selected temperature, to establish the energy absorption and the cleavage fracture versus temperature curves shown in Figure 8 and Figure 9 for low and medium carbon steel, respectively.

Figures 8 and 9 show that uranium exerts a strong beneficial effect in raising the energy absorption curve in the ordinate direction. This is interpreted as being a direct result of the formation of globular sulphides and the consequent reduction of "inclusion fibre" or inclusion anisotropy. This worthwhile benefit is offset to some extent by the tendency of uranium to progressively and markedly raise the fracture-

appearance transition temperature. The effect of uranium on the C_V 15 ft-lb transition temperature is too marginal to detect.

The results shown by Figures 8 and 9 have been confirmed by results obtained from a subsequent series of ultra high sulphur steels treated with uranium.

Longitudinal Impact Tests

Longitudinal Charpy V-notch specimens were tested from all heats in the factorial series. Twenty-four test bars were prepared from a midlength portion of each length of 1/2-inch plate. The test bars were numbered systematically so that they could be selected randomly for subsequent testing to avoid any consistent bias in the results related to position of the test bar in the plate. This was necessary because the plate was only four inches wide and there was a possibility that there would be a significant variation of impact properties across the plate. Also, no test bars were located within one-half inch of the edge of any plate.

The impact bars were broken in groups of three or two over series of temperatures, which allowed a reasonable determination of the average impact energy and fracture transition temperature curves for each steel.

The individual impact results are assembled in Tables 7 and 8. These individual results were averaged and used to plot the impact energy absorption and fracture appearance transition temperatures shown in Figures 10, 11 and 12.

The latter figures illustrate the inability of the classical approach, of varying one factor at a time, to unequivocally determine the effect of uranium and the effect of the presence of sulphur and carbon on the effect of uranium.

Figure 10 shows the effect of uranium on impact transition curves in low carbon steel at three sulphur levels. Note that the curves are not always arranged in the alphabetical sequence corresponding to progressive increases of uranium content. Moreover, the magnitude of the effects of uranium varies with the sulphur level. This apparent inconsistency and variability complicates direct interpretation of the results.

Similarly, Figure 11 shows the effect of uranium on impact transition curves in medium carbon steel at three sulphur levels. In these steels the effect of increasing uranium is always progressive but the effect varies in magnitude at the different sulphur levels.

Figure 12 shows the effect of sulphur on the absorption of impact energy at the two carbon levels and four uranium levels. It may be noted that the effect of sulphur appears to be inconsistent and variable.

The curves of Figures 10, 11 and 12 suggest interaction effects largely because of the underlying design. The chances are that classical design would have required most of the twenty-four heats to determine only the effect of uranium as accurately as the actual factorial assessed the effects of uranium and incidentally the effects of sulphur and carbon. This is so because it is a characteristic of factorial design that, in the absence of interaction, every result is used in the calculation of the effect of each variable just as though the other variables did not exist. This feature is termed "hidden replication".

Prior to statistical analysis, the C_v 15 ft-lb and 50% cleavage transition temperatures were read from the graphs used to produce Figures 10, 11 and 12. These temperatures were then coded by subtracting them from 100 to give positive numbers for analysis. The transition temperature data are given in Tables 8 and 9.

The results of analysis of variance are summarized in Tables 10 and 11. In these tables it will be noted that log per cent sulphur rather than per cent sulphur is one of the main factors or sources of variation. The nominal sulphur levels were, in fact, assigned at equal intervals along a logarithmic scale. The uranium levels were assigned at equal intervals along an arithmetic scale. Consequently, because all of the data was quantitative, it was possible to apply orthogonal coefficients to any factor involving log sulphur or uranium and so to determine, where applicable, the statistical significance of linear, quadratic and cubic components of variance. Knowledge of the significance of these components facilitates the fitting of mathematical expressions to the experimental data.

As well as telling which factors are statistically significant, statistical analysis also gives an estimate of the variability of the experiment itself. This is of some importance in relation to practical and commercial significance. After all, statistical significance is often meaningless or misleading when an experiment is so sensitive that it detects very small effects which have no practical or commercial significance. In Tables 10 and 11 the "pooled" interaction residual mean square is a measure of the random and uncontrolled variance of the experiment. The square root of this variance estimate is known as the standard deviation of each individual experimental result. It happens, as anticipated, that the standard deviation of each individual transition temperature is relatively high and it follows that statistical significance will be related to practical significance in this small experiment. As a corollary it may be pointed out that because the standard deviation is high and the experiment is small the lack of statistical

TABLE 6

Raw Longitudinal Charpy V-Notch Impact Data
(ft-lb and per cent cleavage) for Low Carbon Steel

Steel	Temperature, °C																	
	+80		+60		+40		+27		0		-20		-40		-60		-80	
	ft-lb	%Cl	ft-lb	%Cl	ft-lb	%Cl	ft-lb	%Cl	ft-lb	%Cl	ft-lb	%Cl	ft-lb	%Cl	ft-lb	%Cl	ft-lb	%Cl
A					202	0	206	0	216	0	188	0	174	0	26	95	4	97
					208	0	210	0	222	0	202	0	117	50	10	98	3	97
					216	0			224	0			181	0			3	97
B					209	0	211	0	208	0	108	60	104	60	6	98	4	100
					210	0	202	0	205	0	108	60	95	70	10	96	4	100
					210	0			202	0			73	80	14	96		
C					213	0	52		20	90	5	97	2	100				
					190	0	34		17	90	4	97	2	100				
					220	0			14	95			2	100				
D			94	25	86	25			36	80	15	90	2	98	2	100		
			120	8	80	25			36	80	18	90	4	98	2	100		
					74	40			34	80			2	98				
E					121	0	110	0	113	0	50	55	20	80	6	98	2	100
					97	0	100	0	96	0	92	5	35	80	8	98	2	100
					98	0			102	0			36	75			2	100
F					203	0	184	0	169	0	102	50	63	80	7	98	2	100
					196	0	156	0	151	0	122	40	-	80	6	98	2	100
					197	0			194	0			90	70			3	100
G					185	0	188	0	210	0	170	0	93	70	8	98	2	100
					180	0	178	0	177	0	96	50	90	70	8	98	2	100
					178	0			190	0			12	95			2	100
H	120	10			88	5	55		21	90	10	95	3	98	2	100		
	92	0			104	25	52		20	90	16	94	3	98	2	100		
	140	5			82	50			38	80	18	90						
I			56	0	56	0			56	0	49	1	18	75	9	90	6	100
			58	0	52	0			54	0	49	6	18	75	4	90	1	100
					54	0			55	0			20	70			1	100
J			86	0	94	0			72	10	68	20	34	60	4	95	2	100
			98	0	80	0			70	0	55	10	24	65	6	95	2	100
					84	0			86	0							2	100
K			68	0	73	0			64	2	66	2	22	70	13	90	3	100
			74	0	66	0			62	2	63	1	29	65	10	90	2	100
					68	0			67	1							2	100
L			81	0	83	0			76	15	53	75	28		10	95	5	100
			76	0	80	0			68	15	56	65	24		10	95	4	100
					86	0			72	15			28				3	100

TABLE 7

Raw Longitudinal Charpy V-Notch Impact Data
(ft-lb and per cent cleavage) for Medium Carbon Steel

Steel	Temperature, °C																	
	+100		+80		+60		+40		+27		0		-20		-40		-60	
	ft-lb	%C1	ft-lb	%C1	ft-lb	%C1	ft-lb	%C1	ft-lb	%C1	ft-lb	%C1	ft-lb	%C1	ft-lb	%C1	ft-lb	%C1
M			75	0	66	1	54	40			27	95	13	98	6	100		
			70	0	66	5	50	40			24	95	10	98	6	100		
			76	0			44	45			24	95	16	98	7	100		
N			74	0	58	25	46	50			23	95	17	98	6	100		
			73	1	64	20	47	50			24	95	16	98	6	100		
			72	1			47	50			26	95	15	95	6	100		
O	60	10	56	30	34	60	32	75			12	97	6	99	4	100		
	60	10	54	30	36	60	30	75			12	97	6	99	2	100		
			57	30			30	75			12	97			2	100		
P	41	35	36	50					18	95	6	95	4	100	2	100	1	100
	40	35	34	50					14	95	10	95	3	100	2	100	1	100
									18	95	8	95			2	100	1	100
Q			50	0	46	0	46	0	44	10	22	75	14	90	7	100		
			48	0	48	0	44	0	42	10	21	75	14	90	6	100		
							49	0	46	5	22	75			6	100		
R			58	0	56	1	36	50	32	60	20	85	10	99	6	100		
			60	0	54	5	36	40	28	60	17	90	8	99	6	100		
							35	50	32	60	20	85			7	100		
S			56	20	36	50	28	60	23	90	13	97	4	98	2	100		
			52	20	38	50	28	60	20	90	11	97	6	98	2	100		
							21	70	23	90	8	97			2	100		
T	38	10	38	15	29	40	22	70	18	85	8	95	4	100				
	38	10	34	15	-	15	23	70	16	85	10	95	4	100				
			36	15			22	70	18	85	8	95						
U							31	0	30	0	30	0	20	45	8	85	6	97
							32	0	30	0	32	0	22	45	8	85	6	97
							31	0	33	0	32	0	20	45	10	85	6	100
V							32	0	34	0	31	3	16	75	7	90	6	99
							31	0	34	0	30	3	16	75	9	90	5	99
							33	0	34	0	31	3	16	75	9	90	8	99
W							36	0	35	2	20	65	14	85	6	95	3	100
							35	0	36	2	20	65	12	85	8	95	2	100
							36	0	32	10	21	65	14	85	6	95	5	100
X					50	2	38	20			22	85			7	98	3	100
					49	1	40	25			20	85			10	98	5	100
					48	0	38	0			19	85			8	98	3	100

TABLE 8

Longitudinal C_v 15 ft-lb Transition Temperatures,
Adjusted* Internally for Variance of Carbon
Content and Coded for Analysis of Variance

Steel	C, %	Actual T. T., °C	Adjustment to T. T., °C	Adjusted T. T., °C	Coded T. T. (100 - Adjusted T. T.), °C
A	0.14	-61	-1	-62	162
B	0.15	-55	-2	-57	157
C	0.15	-2	-2	-4	104
D	0.14	-21	-1	-22	122
E	0.13	-46	+1	-45	145
F	0.14	-56	-1	-57	157
G	0.12	-46	+2	-44	144
H	0.13	-17	-1	-16	116
I	0.14	-44	-1	-45	145
J	0.12	-49	+2	-47	147
K	0.11	-53	+6	-47	147
L	0.16	-53	-4	-57	157
M	0.38	-18	-2	-20	120
N	0.36	-18	+1	-17	117
O	0.38	+7	-2	+5	95
P	0.39	+23	-3	+20	80
Q	0.38	-11	-2	-13	113
R	0.39	-9	-3	-12	112
S	0.37	+9	0	+9	91
T	0.39	+20	-3	+17	83
U	0.34	-29	+4	-25	125
V	0.37	-21	0	-21	121
W	0.35	-13	+3	-10	110
X	0.33	-13	+6	-7	107

* Analysis of variance of the unadjusted transition temperatures showed that the transition temperature increased an average of 1.54°C (2.77°F) per 0.01% carbon. Accordingly, the actual transition temperatures were arbitrarily adjusted to correspond to the average carbon levels of either 0.136 or 0.369% carbon.

TABLE 9

Longitudinal C_v 50% Cleavage Transition
Temperatures, Adjusted* Internally for Variation
of Carbon Content and Coded for Analysis of Variance

Steel	C, %	Actual T. T., °C	Adjustment to T. T., °C	Adjusted T. T., °C	Coded T. T. (100 - Adjusted T. T.), °C
A	0.14	-45	-1	-46	146
B	0.15	-25	-3	-28	128
C	0.15	+17	-3	+14	86
D	0.14	+28	-1	+27	73
E	0.13	-27	+1	-26	126
F	0.14	-23	-1	-24	124
G	0.12	-30	+3	-27	127
H	0.13	+23	+1	+24	76
I	0.14	-34	-1	-35	135
J	0.12	-34	+4	-30	130
K	0.11	-34	+5	-29	129
L	0.16	-19	-5	-24	124
M	0.38	+37	-2	+35	65
N	0.36	+40	+2	+42	58
O	0.38	+63	-2	+61	39
P	0.39	+80	-4	+76	24
Q	0.38	+12	-2	+10	90
R	0.39	+35	-4	+31	69
S	0.37	+53	0	+53	47
T	0.39	+56	-4	+52	48
U	0.34	-22	+6	-16	116
V	0.37	-13	0	-13	113
W	0.35	+8	+4	+12	88
X	0.33	+24	+7	+31	69

* Analysis of variance of the unadjusted transition temperatures showed that the transition temperature increased an average of 2.07°C (3.73°F) per 0.01% carbon. Accordingly, the actual transition temperatures were arbitrarily adjusted to correspond to the average carbon levels of either 0.136 or 0.369% carbon.

TABLE 10

Summary of Analysis of Variance of the Longitudinal C_v 15 ft-lb
Transition Temperature Data, from Table 9

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares	F Ratio	Significance Level, α	100 (1 - α)*, %
Uranium	3	2,982.4584	994.1528	11.50	0.005	99.5
(linear)	(1)	(2,566.8750)	2,566.8750	29.68	0.005	99.5
(quadratic)	(1)	(30.3750)	30.3750	<1	not significant	-
(cubic)	(1)	(385.2083)	385.2083	4.45	0.05	95.0
log Sulphur	2	834.3334	417.1667	4.82	0.025	97.5
(linear)	(1)	(650.2500)	650.2500	7.52	0.025	97.5
(quadratic)	(1)	(184.0833)	184.0833	2.13	0.25 (not significant)	-
Carbon	1	7,668.3750	7,668.3750	88.69	0.005	99.5
U x log S	6	1,523.6666	253.9444	2.14	0.25 (not significant)	-
(linear U x linear log S)	(1)	(1,022.4500)	1,022.4500	11.82	0.005	99.5
(linear U x quadratic log S)	(1)	(12.5000)	12.1500	<1	not significant	-
(quadratic U x linear log S)	(1)	(0.2500)	0.2500	<1	not significant	-
U x C	3	95.4580	31.8193	<1	not significant	-
log S x C	2	75.0000	37.50	<1	not significant	-
U x log S x C	6	711.6670	118.611			
Pooled Interaction Residual	(16)	(1,383.3416)	86.4588			
Total	23	13,890.9584				

* 100 (1 - α) is the percentage confidence level or probability that a true effect or result has been detected.

TABLE 11

Summary of Analysis of Variance of the Longitudinal C_v 50% Cleavage
Transition Temperature Data, from Table 8

Source of Variation	Degrees of Freedom	Sums of Squares	Mean Squares	F Ratio	Significance Level, α	100 (1 - α)*, %
Uranium	3	6,832.5000	2,277.5000	14.3	0.005	99.5
(linear)	(1)	(6,720.0333)	6,720.0333	42.2	0.005	99.5
(quadratic)	(1)	(88.1666)	88.1666	<1	not significant	-
(cubic)	(1)	(24.3000)	24.3000	<1	not significant	-
log Sulphur	2	5,324.0834	2,662.0417	16.7	0.005	99.5
(linear)	(1)	(5,076.5625)	5,076.5625	31.9	0.005	99.5
(quadratic)	(1)	(247.5208)	247.5208	1.5	0.25 (not significant)	-
Carbon	1	13,920.1667	13,920.1667	87.5	0.005	99.5
U x log S	6	687.5000	114.5416	<1	not significant	-
(linear U x linear log S)	(1)	(644.1125)	644.1125	4.05	0.10	90.0
U x C	3	131.1667	43.7222	<1	not significant	-
log S x C	2	834.0833	417.0416	1.58	$\alpha = 0.3$ (not significant)	-
U x log S x C	6	1,580.5833	263.4305			
Pooled Interaction						
Residual	(16)	2,545.8333	159.1146			
Total	23					

* 100 (1 - α) is the percentage confidence level or probability that a true effect or result has been detected.

significance of any factor may only mean that more experimentation is necessary to demonstrate the significance or lack of significance of that factor.

In this experiment the respective standard deviations were 9.3°C (16.7°F) and 12.6°C (22.7°F) with sixteen degrees of freedom for the C_v 15 ft-lb and 50% cleavage transition temperatures. This information was used to construct Table 12 which shows the variability in terms of the 95% confidence interval for any true individual or relevant mean value; and, the 95% least significant difference between two individual or relevant mean values. This table illustrates how very variable experimental determinations of transition temperatures may be. In comparing two transition temperatures the difference between them must exceed the tabulated least significant difference if the transition temperatures are statistically different at the chosen tabulated confidence level.

TABLE 12

Relevant 95% Confidence Limits and Least Significant Differences
Applicable to the Experimental Transition Temperatures

Experimental Result	95% Confidence Limits, \pm °C	95% Least Significant Difference, °C
C_v 15 ft-lb T. T.:		
Individual	19.7	27.9
Mean of 2	13.9	19.7
Mean of 3	11.4	16.1
Mean of 4	9.9	13.9
Mean of 6	8.1	11.4
Mean of 8	7.0	9.9
Mean of 12	5.7	8.1
C_v 50% Cleavage T. T.:		
Individual	26.7	37.8
Mean of 2	18.9	26.7
Mean of 3	15.4	21.8
Mean of 4	13.4	18.9
Mean of 6	10.9	15.4
Mean of 8	9.5	13.4
Mean of 12	7.7	10.9

The quantitative treatment effects of uranium, sulphur and carbon are perhaps most self evident in graphical form. Accordingly, the mathematically isolated actual mean treatment effects are shown graphically in Figures 13 to 16 inclusive. Reference to these figures will readily indicate whether or not the statistically significant effects may also have practical significance or magnitude. The captions state which effects are statistically significant. The captions also state whether the plotted points are means of 2, 3, 4, 6, 8 or 12 individual transition temperatures so that by reference to Table 12 the variability of the results may be made more evident and the statistical significance of each treatment effect mean may be assessed.

Figures 13 to 16 inclusive, when interpreted with reference to Tables 10, 11 and 12, show that uranium, sulphur and carbon have exercised commercially significant, as well as statistically significant, control over Charpy impact performance.

Considering the C_V 15 ft-lb transition temperature, uranium has a linear and cubic detrimental effect, log sulphur has a linear (and possibly a quadratic) effect which is beneficial at the 0.30 and 0.65% uranium levels, and carbon has a detrimental effect. A third level of carbon would be necessary to determine the nature of the curve describing the effect of carbon.

Considering the C_V 50% cleavage transition temperature, uranium has a linear detrimental effect and log sulphur has a linear beneficial effect. Carbon has a detrimental effect.

With both transition temperatures there was a linear uranium by sulphur interaction. The actual effect of uranium depends not only on the level of uranium but also on the level of sulphur. Conversely, the effect of sulphur depends not only on the level of sulphur but also on the level of uranium. The interaction may be said to be self-mitigating and beneficial since increasing sulphur decreases the harmful effects of uranium, while conversely, increasing uranium tends to increase the beneficial effect of sulphur.

Figure 13 shows the mean treatment effects of nil, 0.14, 0.30 and 0.65% uranium at sulphur levels of 0.006, 0.32 and 0.139% on the C_V 15 ft-lb and 50% cleavage transition temperatures. Figure 13 (left) illustrates the finding of the analysis of variance that the relationship between uranium level and C_V 15 ft-lb transition temperature requires a third order equation with a linear uranium by linear log sulphur interaction term. Figure 13 (right) illustrates the finding of the analysis of variance that the relationship between uranium level and C_V 50% cleavage requires a linear equation with a linear uranium by linear log sulphur interaction term. The C_V 50% cleavage transition temperature increased at a rate of 7.5°C (13°F) per 0.10% uranium.

Figure 14 shows the mean treatment effects of nil, 0.14, 0.31 and 0.645% uranium at carbon levels of 0.136 and 0.369% on the C_V 15 ft-lb and C_V 50% cleavage transition temperatures. The curves illustrate the absence of a uranium by carbon interaction. Because of this absence the curvilinear effect of uranium is more clearly illustrated than in Figure 13.

Figure 15 shows the mean treatment effects of 0.006, 0.032 and 0.139% sulphur at uranium levels of nil, 0.14, 0.30 and 0.65%. Figure 15 (left) illustrates the finding of the analysis of variance that the relationship between log sulphur and C_V 15 ft-lb transition temperature requires a second-order equation with a linear uranium by linear log sulphur interaction term. The curves indicate that, in the absence of uranium, sulphur is either innocuous or slightly beneficial to the C_V 15 ft-lb transition temperature. However, statistically the effect of sulphur, in the absence of uranium, was not significant. Sulphur is always beneficial to the C_V 50% cleavage transition temperature. Sulphur, in the absence of uranium, lowers the C_V 50% cleavage transition temperature by about 2.0°C (3.5°F) per 0.01% sulphur.

Figure 16 shows the mean treatment effects of 0.136 and 0.369% carbon at uranium levels of nil, 0.14, 0.30 and 0.65% on the C_V 15 ft-lb and 50% cleavage transition temperatures. The plotted points are tightly grouped, indicating that there is no uranium by carbon interaction. It is assumed that the relationships between carbon and the transition temperatures are linear. On this assumption, carbon may be said to raise the C_V 15 ft-lb transition temperature by an average of 1.5°C (2.7°F) per 0.01% carbon; and to raise the C_V 50% cleavage transition temperature by an average of 2.0°C (3.6°F) per 0.01% carbon.

Figure 17 shows the effect of carbon at each sulphur level (left) and the effect of sulphur at each carbon level (right) on the C_V 50% cleavage transition temperature. The existence of the apparent sulphur by carbon interaction is not adequately supported by the analysis of variance. There is only a two-to-one probability ratio that the interaction did not occur by chance.

GENERAL DISCUSSION

When this project was planned it was not anticipated, with any reasonable confidence based on available metallurgical data and previous experimental results, that uranium would be found to have much merit as an alloying element in steel. Accordingly, the project was planned not only to have inherent merit in phasing out much of the uranium program

by re-evaluating some of the controversial results, but also to produce some results of more positive significance. Thus, the factors of sulphur and carbon became important in themselves, and in addition some emphasis was placed on demonstrating some of the advantages of using certain statistical design and analysis techniques rather than conventional ad hoc (try it once or twice and see) and classical (vary one factor at a time) techniques.

The influence of uranium in low and medium wrought carbon steels has now been evaluated by study of numerous important properties of as-rolled low and medium carbon steels. For each such property, except machinability, it has been demonstrated conclusively whether or not uranium has an innocuous, a marginal or a large but highly specialized influence of restricted commercial importance. Uranium has been shown to refine the as-cast macrostructure but not to affect the as-rolled ferritic grain size. Uranium has been shown to inhibit and slow the isothermal transformation of austenite, and to affect the morphology of pearlite and ferrite, but to have virtually no effect on tensile properties. Uranium has been shown to modestly lower active-state corrosion rates in 5% hydrochloric acid and in 10% sulphuric acid; to greatly reduce end-grain pitting, especially in resulphurized steel; and to improve resistance to stress-corrosion cracking in the I.G. Farben test. Uranium has been shown to have a strong affinity for sulphur and to control completely the morphology of sulphides. Constant-pressure lathe tests did not indicate that such control of the sulphides affected machinability. It is still considered possible that production-run machinability tests might yield favourable results. Uranium, by control of sulphides, has been shown to improve energy absorption greatly in transverse impact tests. In longitudinal impact tests, uranium is innocuous below 0.2% and deleterious above 0.3% to the C_v 15 ft-lb transition temperature. Uranium is always detrimental to fracture appearance transition temperature. Invariably, the deleterious effects of uranium are mollified by the presence of sulphur.

The above findings of this investigation are considered to be independent of effects of macrosegregation of uranium-rich inclusions, by virtue of the type of ingot used and by virtue of applied statistical procedures.

The effects of sulphur on impact properties have been reported for carbon steels by Rinebolt and Harris^(28, 29) and for alloy steels by Hodge, Frazier and Boulger⁽³⁰⁾. It is generally known that sulphur has a marked and progressively deleterious effect on impact energy absorption of cast steels. The results of Rinebolt and Harris were somewhat indeterminate because of wide scatter which they attributed to laminations caused by the sulphides. Hodge, Frazier and Boulger found that the effect of sulphur in decreasing maximum energy absorption was related linearly to the log

per cent sulphur; that neither the fracture transition temperature (average energy) nor the ductility transition temperature was markedly affected by the sulphur level, although sulphur appeared to be beneficial at high levels; and that there was a tendency for sulphur to promote fibrous rather than brittle fracture. The findings of Hodge, Frazier and Boulger for heat-treated steels appear now to be corroborated for carbon steels, and it has been found that sulphur definitely promotes the appearance of fibrous rather than cleavage fracture, as shown in Figure 15 (right).

At all levels uranium is detrimental and sulphur is beneficial to the C_v 50% cleavage transition temperature. This transition temperature is important because it relates to the manner and ease of propagation of fracture. The fracture transition temperature is sensitive to conditions existing at the leading edge of the fracture and is relatively insensitive to notches, changes in loading, and specimen geometry. Thus, anything which lowers the C_v 50% cleavage transition temperature is beneficial because it lowers the service temperature below which fracture propagates catastrophically along cleavage planes. Cleavage fractures are considered or termed catastrophic because they may be propagated by the release of internally stored elastic energy and may travel at speeds approaching about one-third of the velocity of sound in metal, i. e. about 5,000 ft/sec.

The ductility transition temperatures, as exemplified by the C_v 15 ft-lb transition temperature, have great practical significance because they relate in large measure to the ease of initiation of fracture. These transition temperatures, in contrast to fracture transition temperatures, are sensitive to changes in specimen geometry, notch geometry and rate of loading. As the specimen size increases, the degree of triaxiality of stresses increases; as the depth and sharpness of notches increase, the stress concentrations become more severe; and as the rate of loading increases, the strain rate increases. These conditions all effectively raise the temperature at which fracture may readily be initiated. The result is that the margin of safety in engineering structures is often narrow, especially in cold climates. All elements added to steel affect the ductility transition temperatures in some measure and direction. Many common additions to steel raise the ductility transition temperatures sharply. Therefore, control of composition, based on knowledge of the effect of alloying elements on transition temperatures, is of considerable commercial significance.

It is logical to study the influence of alloying elements by the use of statistically designed and analyzed experiments. In planning the necessary experiments it is important to have an estimate of the probable standard deviation so as to optimize the size of the experiment. The technical literature contains very few estimates of the standard deviation of transition temperatures and these are based on steels of carefully

controlled structure. The present report contains some standard deviations based on experimental steels having a greater, and perhaps more realistic, variation in structure. The differences between the reported standard deviations may therefore be of interest to many who plan to investigate transition temperatures in steel.

Rinebolt and Harris^(28, 29) investigated the influence of a number of elements on the impact characteristics of steel having a base composition of 0.30% C, 1.0% Mn, 0.38% Si and 0.005% P. Their steel was carefully normalized, or normalized and tempered, to produce an almost invariant microstructure of relatively coarse pearlite and a ferritic grain size of 7-8. The program was designed in the classical manner. Sixteen carefully matched heats of the base analysis were used for reference or control. The standard deviations of these sixteen controls were 4.5°C (8.2°F) and 7.8°C (14.0°F) respectively for the C_V 15 ft-lb and 50% cleavage transition temperatures. The comparable standard deviations of the present work are 9.3°C (16.7°F) and 12.6°C (22.7°F) respectively for the C_V 15 ft-lb and 50% cleavage transition temperatures. Both of these pairs of standard deviations are based on 15 degrees of freedom and so are directly comparable. It is believed that the greater size of the standard deviations in the present work is accounted for by the wider fluctuation of the chemical compositions, the smaller degree of reduction during hot rolling, and the wider fluctuation of impact properties expected from as-rolled steels. The differences between the standard deviations and the size of each deviation indicate how carefully investigations of impact properties must be conducted and interpreted. The wider set of deviations is probably more relevant to most experimental situations and certainly closer to commercial reality.

The literature contains some estimates of the effect of carbon on various impact transition temperatures. It is difficult to find good agreement among them for the C_V 15 ft-lb transition temperature unless it is assumed that the relationship between increase of carbon and increase of transition temperature is curvilinear. The present work indicates that over the range of 0.14 to 0.37% carbon the C_V 15 ft-lb transition temperature is raised 1.5°C (2.7°F) per 0.01% carbon. This is in exact agreement with Rinebolt and Harris⁽²⁸⁾ over the range of 0.11 to 0.43% carbon. Rinebolt and Harris⁽²⁹⁾ show that the C_V 15 ft-lb transition temperature is raised 1.04°C (1.9°F) and 4.6°C (8.3°F) per 0.01% carbon over the ranges of 0.01 to 0.4% carbon and 0.4 to 0.74% carbon, respectively. Boulger and Hansen⁽³¹⁾, testing ship-plate laboratory steels over the range of 0.10 to 0.32% carbon, found that for constant grain size an increase of 0.01% carbon raised the C_V 15 ft-lb transition temperature an average of 1.85°C (3.33°F). Their data were adjusted for the fact that carbon refines the ferritic grain size and by assuming on reported evidence that an increase of one in the ASTM grain size decreases the C_V 15 ft-lb transition

temperature by 11°C (20°F). A similar adjustment to the present data indicates that for constant grain size an increase of 0.01% carbon raises the C_V 15 ft-lb transition temperature by 1.8°C (3.2°F).

The C_V 50% cleavage transition temperature was found to increase linearly with increase of carbon content at the rate of 2.0°C (3.6°F) per 0.01% carbon. Rinebolt and Harris⁽²⁹⁾ showed a rate of 3.1°C (5.6°F) over the range of 0.01 to 0.74% carbon. Their rate is twice that reported in this investigation and three times as great as that reported by Battelle⁽³²⁾ for two-thirds width transverse Charpy bars of API X-52 line pipe.

CONCLUSIONS

As-rolled low and medium carbon steels have been treated with up to 0.6% uranium and analyzed so as to assess qualitatively or quantitatively many of the previously reported marginal, anomalous and otherwise interesting effects of uranium. This was done by using a type of ingot in which most of the uranium macro-segregates could be removed by bottom cropping, and by using statistical design and analysis to eliminate the possibility of any bias in the results due to macrosegregation of uranium. In addition, the effects of sulphur and carbon on Charpy impact properties have been assessed. Finally, the variability of Charpy impact properties has been assessed for future use in the design of experiments.

Specifically it has been found that:

1. Uranium, in sufficient quantity and depending on the sulphur content, refines the as-cast (austenitic) macrostructure.
2. Uranium does not affect the as-rolled ferritic grain size of carbon steel fully killed by aluminum.
3. Uranium, in sufficient quantity and depending on the sulphur content, promotes the formation of degenerate or divorced pearlite and the formation of veining substructure in ferrite.
4. Uranium weakly delays the isothermal transformation of austenite.
5. Despite the effects noted in the preceding conclusions, uranium has almost no effect on tensile properties.
6. Uranium produces steel relatively free of alumina inclusions.

7. Uranium, in sufficient quantity, controls sulphide morphology.
8. Sulphur, even in small amounts, suppresses the formation of UF_2 and so improves the forgeability of uranium steels.
9. Constant-pressure lathe tests indicate that uranium has no effect on machinability.
10. Steels made from electrolytic iron (very low residual copper) have much higher active-state corrosion rates than steels containing normal commercial amounts of residual elements.
11. Uranium, at low levels, may enhance the resistance of steel to stress-corrosion failure.
12. Uranium, in sufficient quantity, greatly improves the impact energy absorption of transverse specimens of resulphurized steels.
13. In tests based on longitudinal impact specimens:
 - a) The standard deviation of C_V 15 ft-lb transition temperatures was $9.3^\circ C$ ($16.7^\circ F$) with sixteen degrees of freedom.
 - b) The standard deviation of C_V 50% cleavage transition temperature was $12.6^\circ C$ ($22.7^\circ F$) with sixteen degrees of freedom.
 - c) Uranium, above about 0.2%, is detrimental to the C_V 15 ft-lb transition temperature. The relationship is linear and cubic in nature.
 - d) Sulphur is beneficial to the C_V 15 ft-lb transition temperature at the 0.3 and 0.6% uranium levels.
 - e) In the absence of uranium, sulphur beneficially lowers the C_V 50% cleavage transition temperature by $2.0^\circ C$ ($3.5^\circ F$) per 0.01% sulphur.
 - f) Uranium has a linear detrimental effect on the C_V 50% cleavage transition temperature. The rate is $7.5^\circ C$ ($13.0^\circ F$) per 0.10% uranium.
 - g) The effects of uranium and sulphur are interdependent. The interaction is such that sulphur decreases the detrimental effects of uranium while uranium increases the beneficial effects of sulphur.
 - h) Carbon raises the C_V 15 ft-lb transition temperature by an average of $1.5^\circ C$ ($2.7^\circ F$) per 0.01% carbon. Correcting for grain size, the rate is $1.8^\circ C$ ($3.2^\circ F$) per 0.01% carbon.

- i) Carbon raises the C_v 50% cleavage transition temperature by an average of 2.0°C (3.6°F) per 0.01% carbon.

A general conclusion, which appears evident when all of the work with uranium is reviewed, is that if uranium has an economic usefulness in steel it will be specialized and minor. Uranium not only has high intrinsic cost, and prospects for even higher cost, but also its use incurs high production costs because of lower yields and variable recoveries. Furthermore, uranium can only be recovered in fully-killed steels. These disadvantages and others appear to outweigh the improvements in cleanness, corrosion resistance, resistance to stress-corrosion and impact energy absorption which were observed.

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REFERENCES

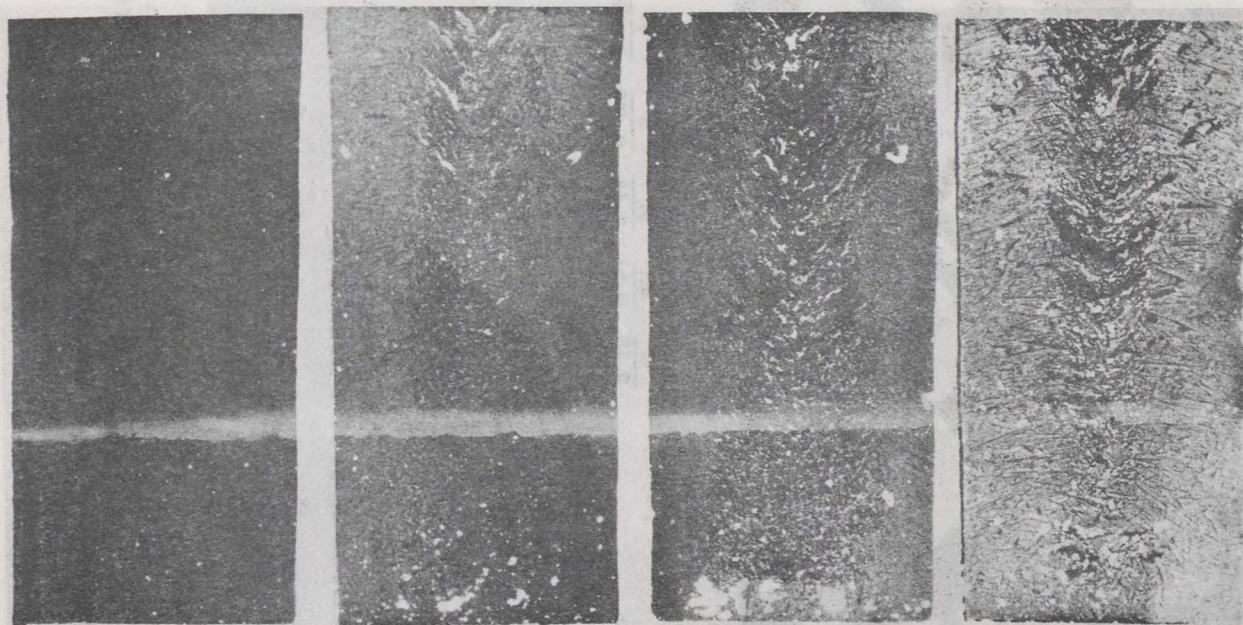
1. Charles E. Makepeace (Eldorado Mining and Refining Limited), "The Non-Atomic Uses of Uranium" - A Bibliography of Metallurgical Abstracts (February 1, 1960).
2. W.A. Morgan, "A Survey and Assessment of Patents Relating to the Use of Uranium in Ferrous Metals" - Internal Report PM-1-60-12 (May 26, 1960).*
3. "Influence of Uranium Additions to Ferrous Alloys - An Interim Review" - Mines Branch Research Report R 95 (April 1962).
Editors: R.F. Knight and D.K. Faurschou.

4. R. D. McDonald, "Stress Corrosion and Hydrogen Embrittlement of Uranium-Bearing Steel" - Internal Reports PM-R-62-21 (October 3, 1962) and PM-R-66-5 (March 15, 1966).*
5. W. J. Wrazej, "The Microstructure and Hardness of Uranium-Bearing Iron and Iron-Carbon Alloys" - Internal Report PM-R-62-23 (October 15, 1962).*
6. R. F. Knight and G. P. Contractor, "The Effect of Uranium on Strain Ageing" - Internal Report PM-R-62-24 (October 16, 1962).*
7. R. F. Knight, G. P. Contractor and D. A. Munro, "Some Observations on Uranium Distribution and Its Effect on the Tensile and Fatigue Properties of Carbon and Carbon-Manganese Steels" - Internal Report PM-R-63-21 (June 7, 1963).*
8. G. J. Biefer and J. G. Garrison, "Exploratory Polarization Measurements on Uranium-Free and Uranium-Bearing Steels" - Internal Report PM-R-63-29 (September 20, 1963).*
9. R. F. Knight and G. P. Contractor, "Transition Temperature of Two Uranium-Bearing Steels" - Internal Report PM-R-63-30 (August 28, 1963).*
10. G. P. Contractor, "Uranium Additions to 0.10% Carbon Steel" - Internal Report PM-R-63-4 (February 15, 1963).*
11. G. P. Contractor and D. A. Munro, "Some Observations on the Tempering Response of Low-Carbon Uranium-Bearing Steel" - Internal Report PM-M-65-6 (April 27, 1965).*
12. G. P. Contractor, "Autoradiographic and Analytical Surveys of Uranium-Bearing Mild Steel Ingots" - Internal Report PM-R-61-10 (March 28, 1961).*
13. R. K. Buhr and G. P. Contractor, "Uranium Segregation Patterns in Ingots Cast at the Physical Metallurgy Division" - Internal Report PM-I-61-10 (June 27, 1961).*
14. R. K. Buhr and D. R. Bell, "Recovery and Segregation of Uranium in Steel" - Internal Report PM-M-62-11 (June 8, 1962).*
15. G. P. Contractor and R. K. Buhr, "Method of Removing Aluminum Oxides from Aluminum-Killed Steels and Steels Produced by Such a Method" - Canada Patent 680,706, February 25, 1964 (U.S.A. Patent 3,119,159; U. K. Patent 954,164).

16. R. Wayne Parcel and Robert F. Mehl, "Effect of Molybdenum and of Nickel on the Rate of Nucleation and Rate of Growth of Pearlite", Trans. AIME, Vol 188, 1952, pp 771-780.
17. C.E. Makepeace (Eldorado Mining and Refining Limited), "The Electrolytic Extraction, Separation, and Identification of Metallic and Non-Metallic Phases in Uranium-Bearing Steel" (Project F-47) - Internal Report PM-R-63-11 (March 15, 1963); Internal Report PM-R-64-14 (May 28, 1964); Internal Report PM-R-64-15 (April 30, 1964).*
18. M.J. Walker, "The Effect of Uranium on the TTT Characteristics of Medium and Low Carbon Steel" - Internal Report PM-S-64-1 (January 8, 1964).*
19. G.J. Biefer, "Exploratory Corrosion Tests on Miscellaneous Uranium-Bearing Ferrous Alloys in Acid Solutions" - Internal Report PM-R-61-9 (April 13, 1961).*
20. M. Hasegawa and I. Onoda, "Additions of Uranium to 0.30-0.40% Carbon Steels" - TETSU-TO-HAGANE (Japanese JISI), Vol 50, Jan. 1964, pp 48-56. (In Japanese).
21. M. Hasegawa and I. Onoda, "Additions of Uranium to Low Carbon Steels" - TETSU-TO-HAGANE (Japanese JISI), Vol 49, Dec. 1963, pp 1788-1799. (In Japanese).
22. E. Williams and M.E. Komp, "Effect of Copper Content of Carbon Steel on Corrosion in Sulfuric Acid" - Corrosion, Vol 21, No. 1, January 1965, pp 9-14.
23. G.P. Contractor and R.D. McDonald, "Uranium-Bearing Steel" - Canada Patent 609,013, November 22, 1960.
24. G.P. Contractor, D.E.C. King and R.J. McClure, "Free-Machining Steels of Improved Transverse Mechanical Properties and Method of Making Same" - Canada Patent 690,748, July 14, 1964 (U.K. Patent 975,337).
25. L.H. Van Vlack, "Correlation of Machinability with Inclusion Characteristics in Resulphurized Bessemer Steels" - Trans. ASM, Vol 45, 1953, pp 741-757.
26. M.E. Merchant and N. Zlatin, "Basic Reasons for Good Machinability of Free-Machining Steels" - Trans. ASM, Vol 41, 1949, pp 647-677.

27. D.W. Murphy and P.T. Aylward, "Machinability of Steel" - Bethlehem Steel Corporation, Booklet 2026 653, undated.
28. J.A. Rinebolt and W.J. Harris, Jr., "Effect of Alloying Elements on Notch Toughness of Pearlitic Steels" - Trans. ASM, Vol 43, 1951, pp 1175-1214.
29. J.A. Rinebolt and W.J. Harris, Jr., "Statistical Analysis of Tests of Charpy V-Notch and Keyhole Bars" - Welding Journal, Vol 30, April 1951, pp 202-s to 208-s.
30. J.M. Hodge, R.H. Frazier and F.W. Boulger, "The Effects of Sulphur on the Notch Toughness of Heat-Treated Steels" - Trans. AIME, Vol 215, October 1959, pp 745-753.
31. F.W. Boulger and W.R. Hansen, "Effects of Metallurgical Variables on Charpy and Drop-Weight Tests" - Trans. AIME, Vol 227, October 1963, pp 1212-1225.
32. Summary Report, "Research on the Properties of Line Pipe" - Battelle Memorial Institute-American Gas Association, Catalogue No. 40/PR, May 1962.

* These are divisional reports of the Mines Branch,
Department of Mines and Technical Surveys, Ottawa, Canada.



0.15 % U

C	Mn	Si	S	P
0.14	0.66	0.030	0.009	0.003

0.34 % U

C	Mn	Si	S	P
0.16	0.62	0.15	0.017	0.011

0.53 % U

C	Mn	Si	S	P
0.13	0.63	0.16	0.022	0.008

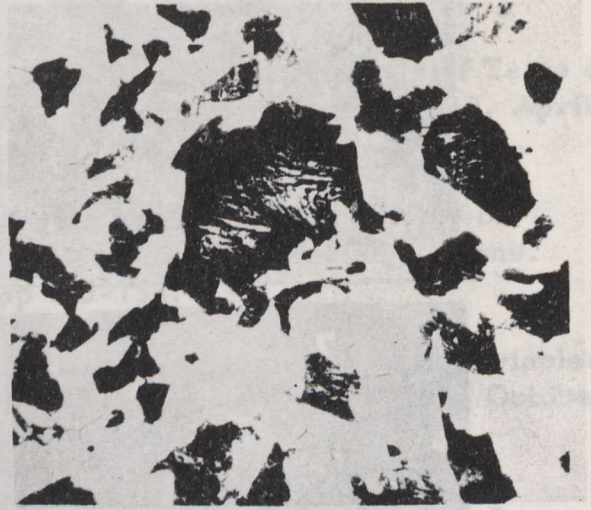
0.76 % U

C	Mn	Si	S	P
0.31	0.63	0.19	0.035	0.006

Figure 1 - Prints of autoradiographs showing the distribution of uranium-rich inclusion clusters (white areas) along the central vertical plane of four representative 4-inch diameter sand-cast billets.

17. E. W. Murphy and P. T. Hayward, "Machinability of Steel" - Bethlehem Steel Corporation, Research 2426-533, undated.

18. J. A. Ringbolt and W. J. Harris, Jr., "Effect of Alloying Elements on Machinability of Pearlitic Steels" - Trans. A.S.M., Vol. 43, 1951.



Steel M		No U		
C	Mn	Si	S	P
0.38	0.65	0.21	0.006	0.002

Steel O		0.37% U		
C	Mn	Si	S	P
0.38	0.61	0.28	0.009	0.006

← 1" →

Figure 2 - Representative microstructures of as-rolled medium carbon steel showing the effect of 0.37% uranium.

Etched in nital. Originally X500 (see scale).

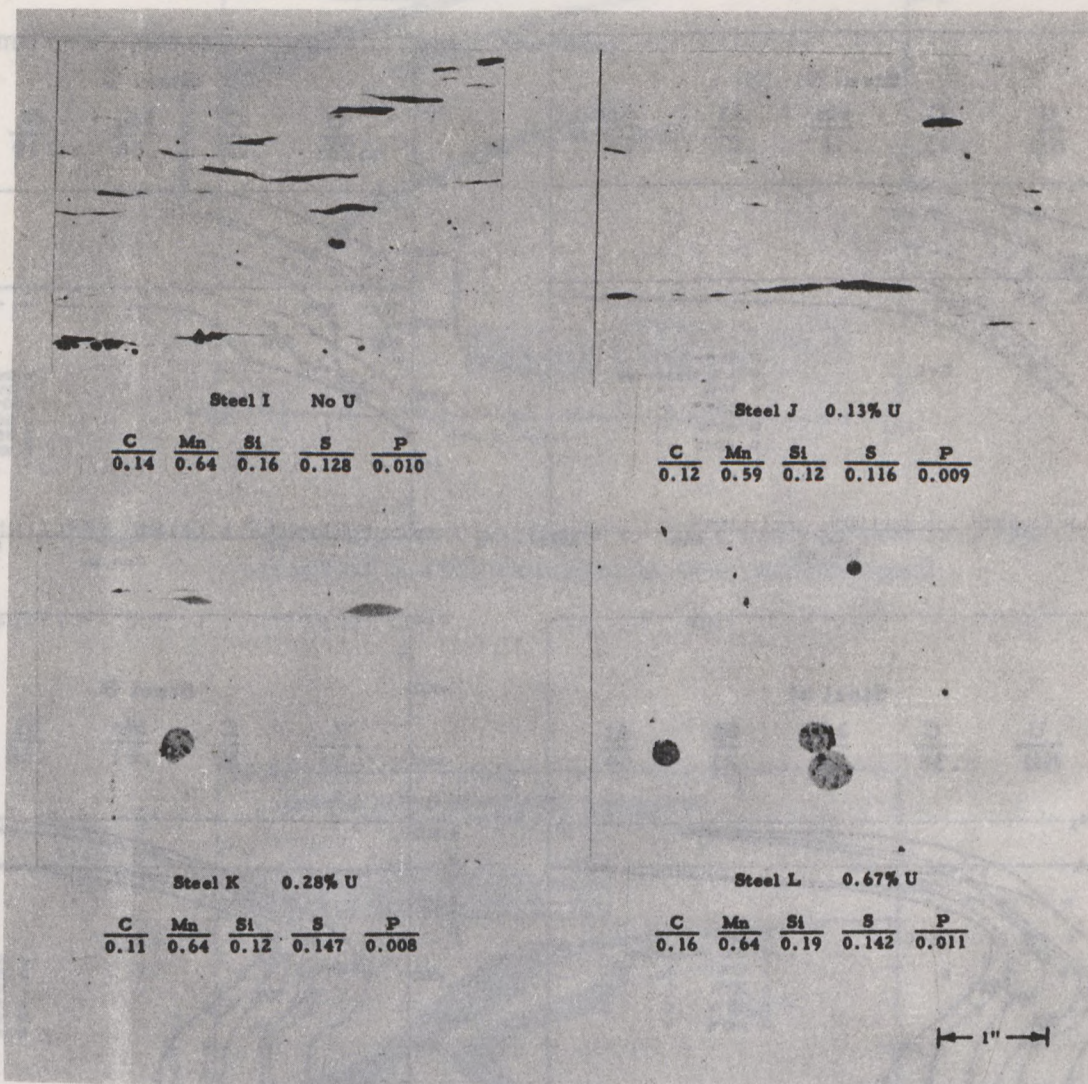


Figure 3 - Representative unetched microstructures showing the effect of 0.13, 0.28 and 0.67% uranium on the morphology of sulphide inclusions in wrought carbon steel.

Originally X500 (see scale).

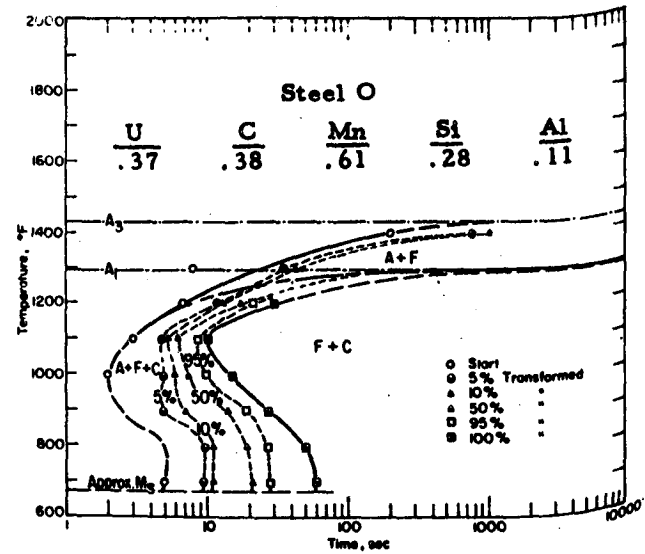
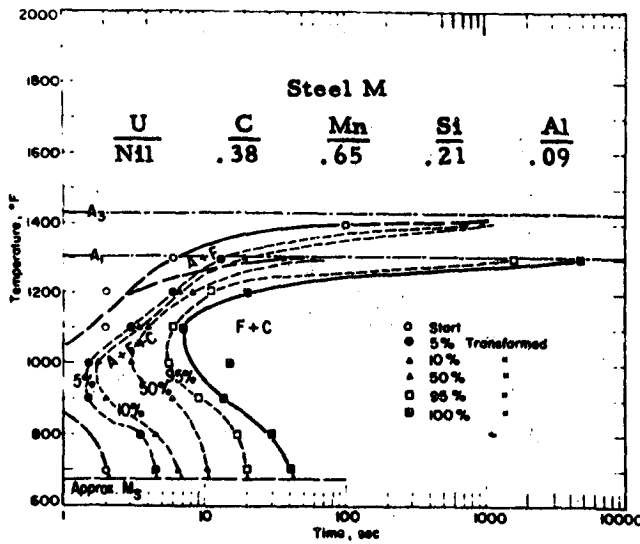
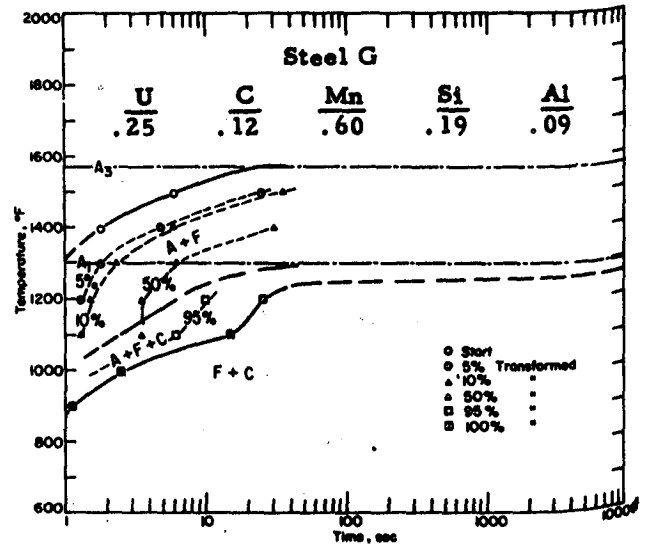
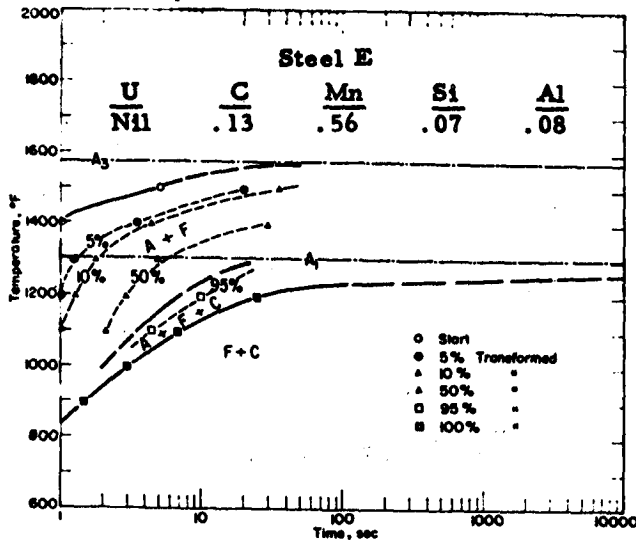


Figure 4 - Isothermal transformation diagrams showing the effect of 0.25% uranium in low carbon steel and of 0.37% uranium in medium carbon steel.

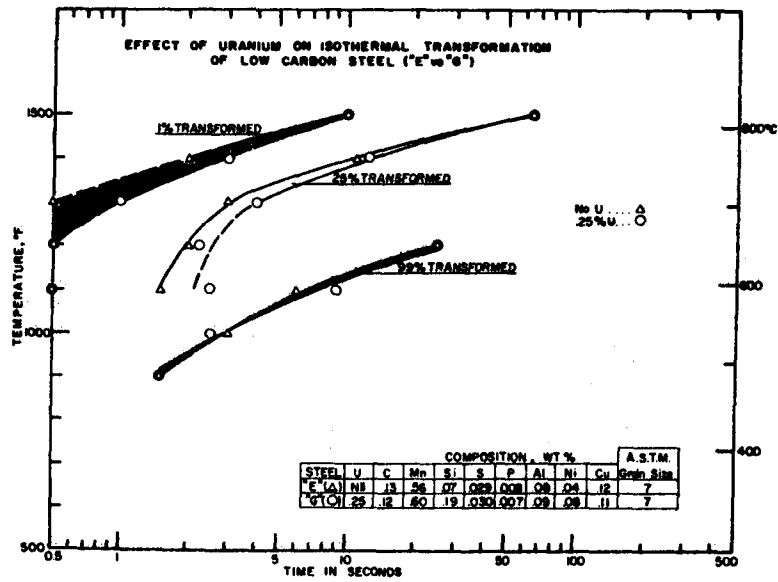


Figure 5 - Superimposed portions of TTT curves showing the effect of 0.25% uranium in low carbon steel.

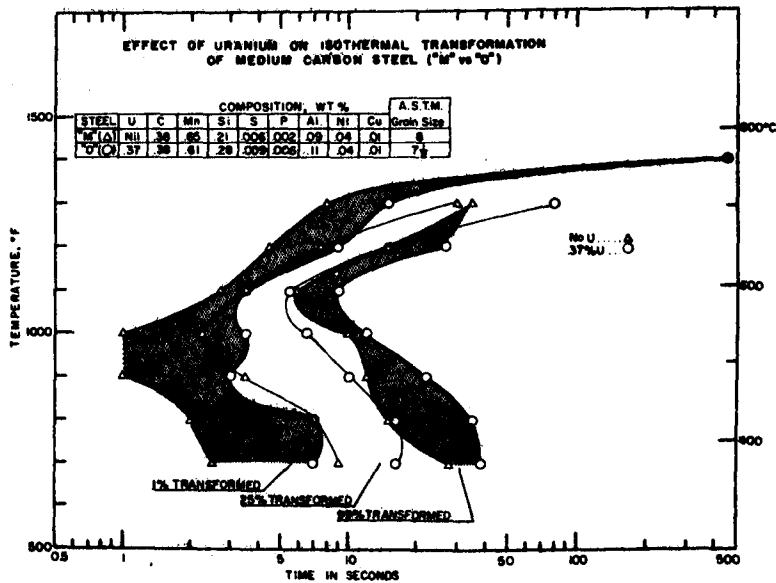


Figure 6 - Superimposed portions of TTT curves showing the effect of 0.37% uranium in medium carbon steel.

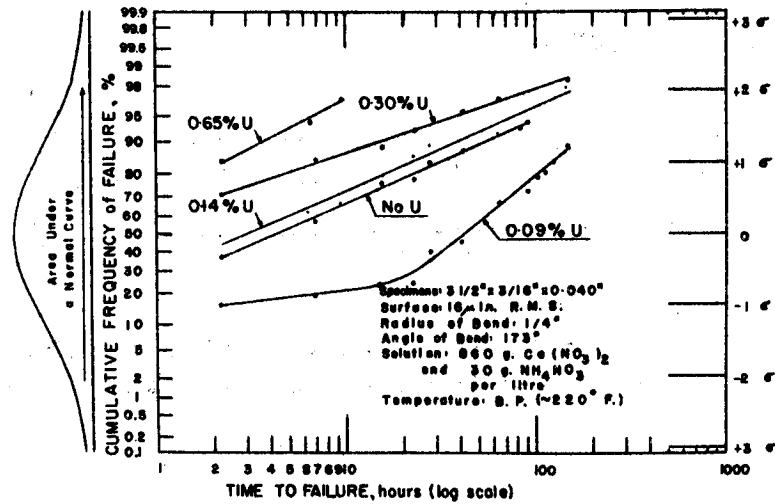


Figure 7 - Results of accelerated stress-corrosion testing of thirty heats of low and medium as-rolled carbon steels in a boiling aqueous calcium nitrate-ammonium nitrate solution, showing the influence of five nominal levels of uranium.

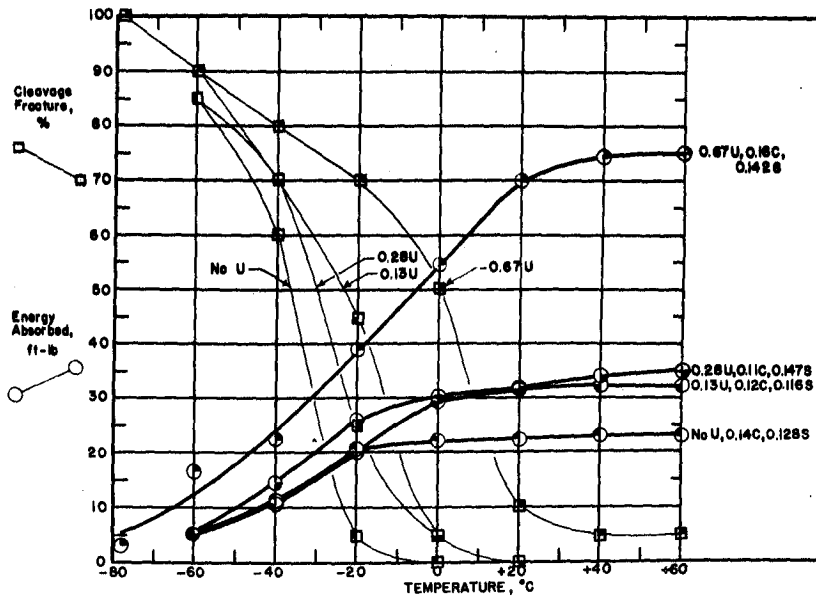


Figure 8 - Ad hoc transverse Charpy V-notch impact transition curves for low carbon steel showing the comparative influence of nil, 0.13, 0.38 and 0.67% uranium.

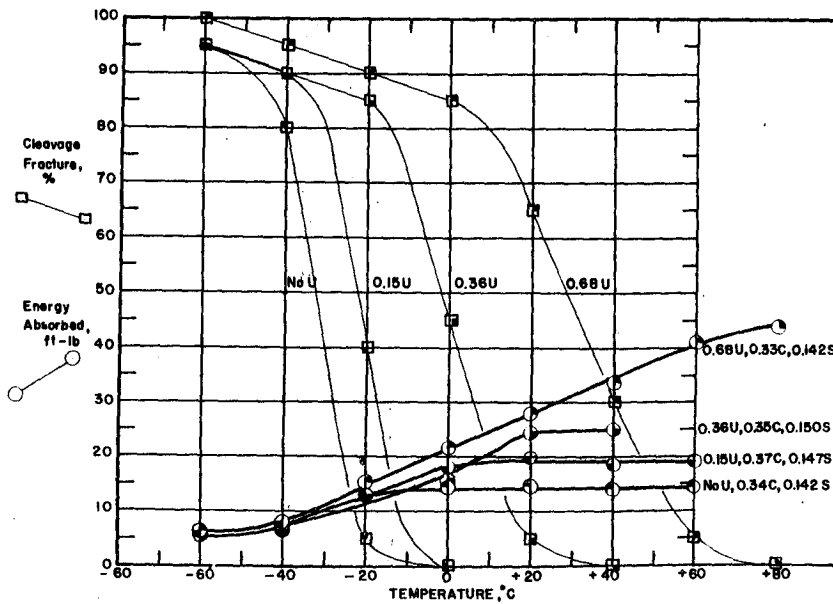
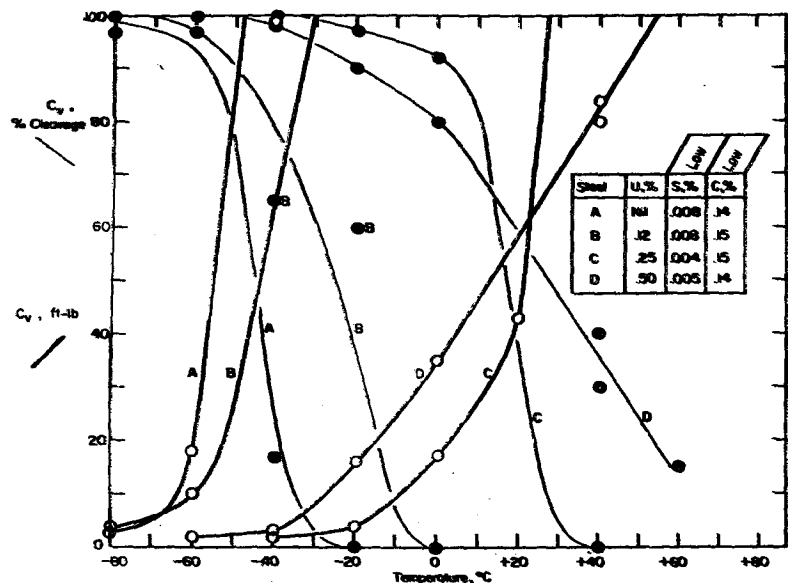
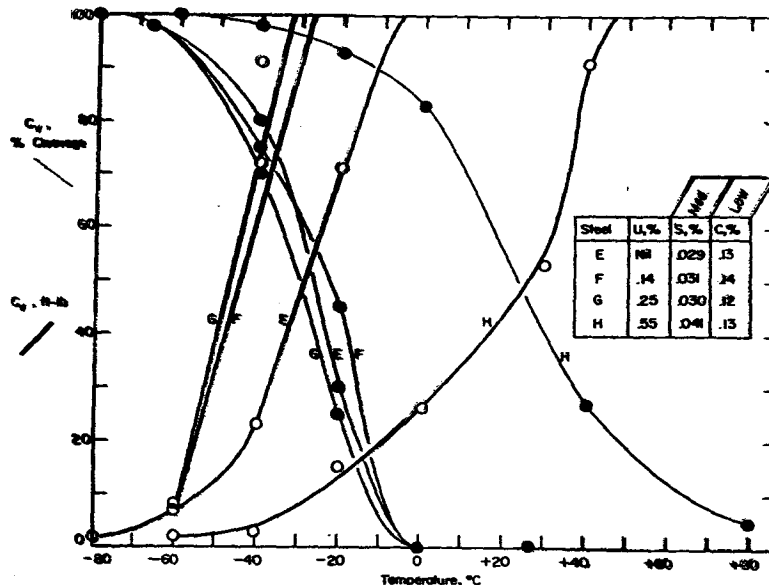


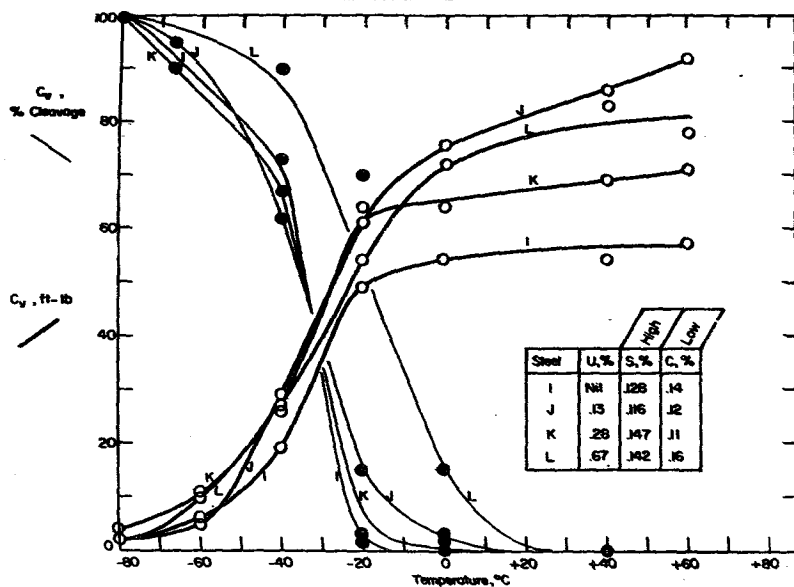
Figure 9 - Ad hoc transverse Charpy V-notch impact transition curves for medium carbon steel showing the comparative influence of nil, 0.15, 0.36 and 0.68% uranium.



Effect Of Uranium On C_v Energy Absorption And Cleavage Fracture At Low S And Low C Levels.



Effect Of Uranium On C_v Energy Absorption And Cleavage Fracture, At Medium S And Low C Levels.



Effect Of Uranium On C_v Energy Absorption And Cleavage Fracture, At High S And Low C Levels.

Figure 10 - Longitudinal C_v energy absorption and fracture appearance transition temperature curves for the twelve low carbon steels. The graphs show the effect of each of four uranium levels at the low sulphur (upper left), medium sulphur (upper right) and high sulphur (lower left) levels.

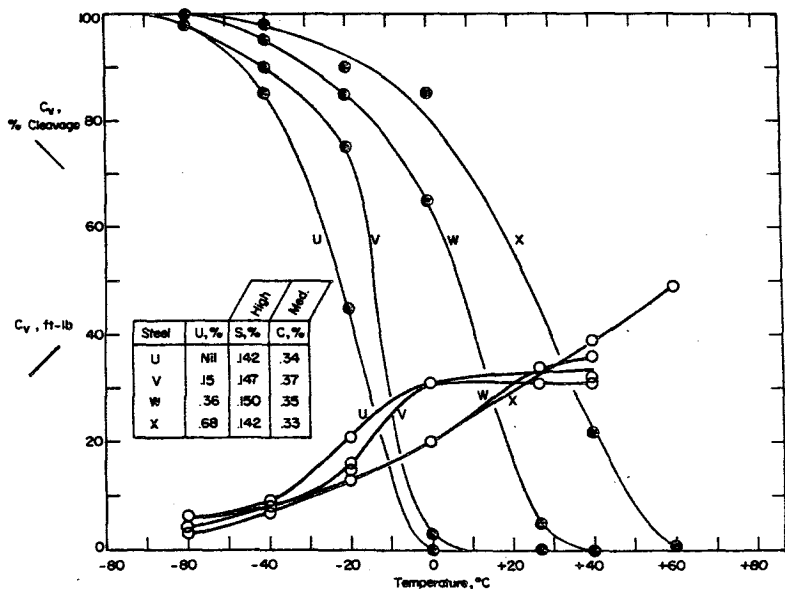
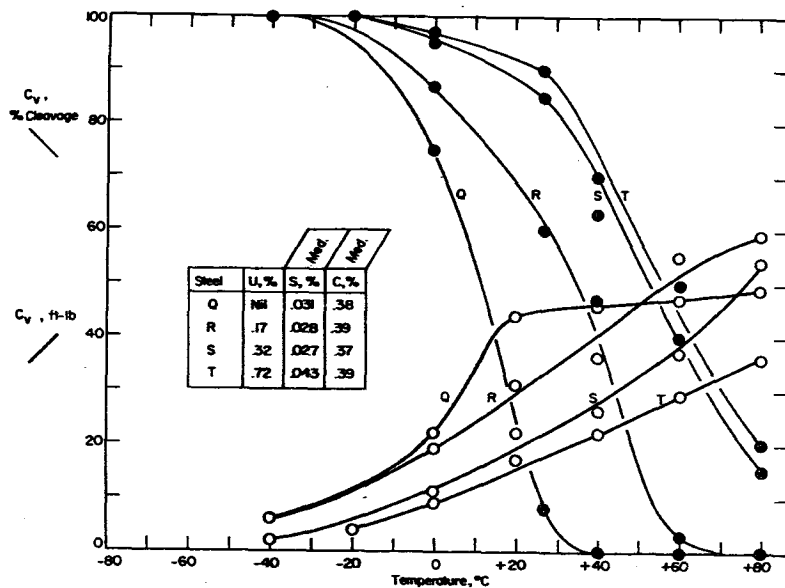
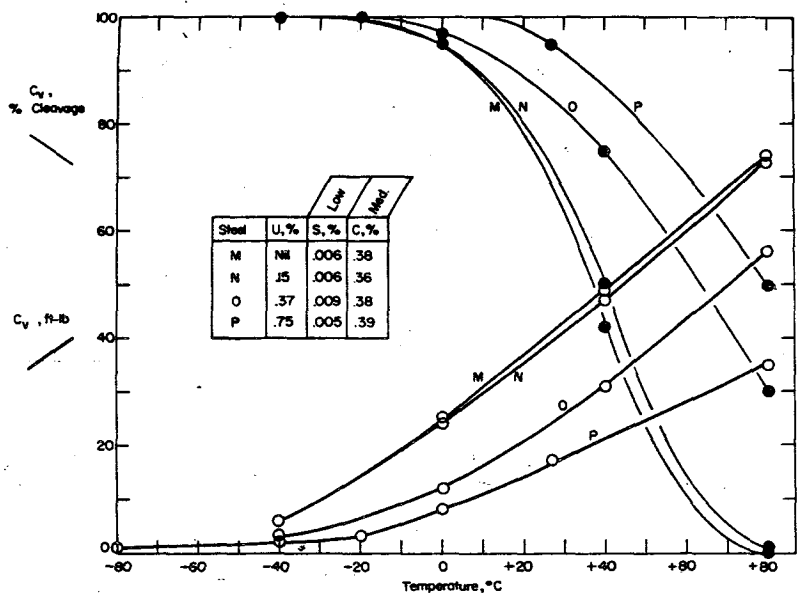
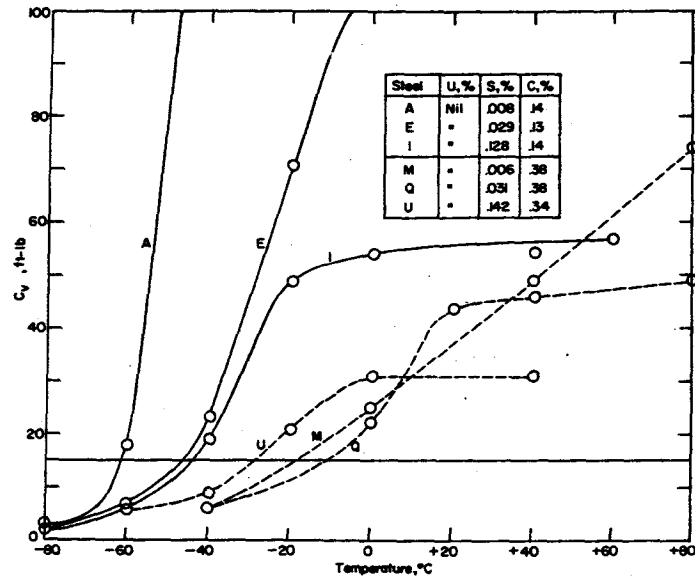
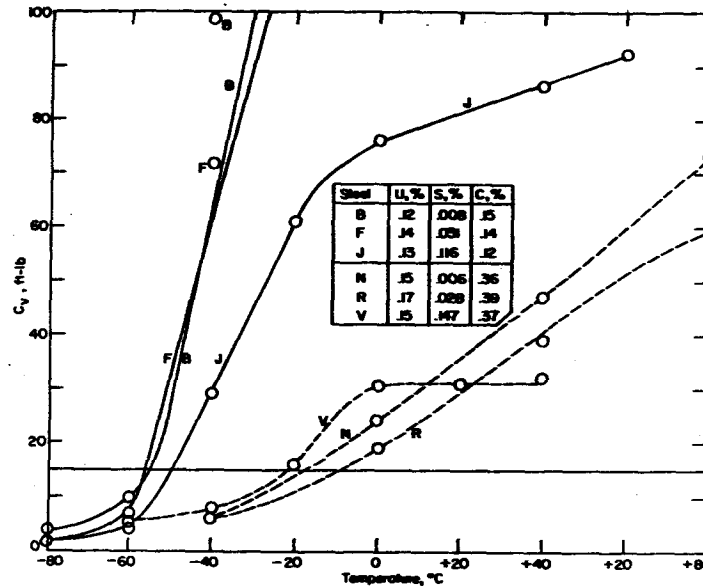


Figure 11 - Longitudinal C_v energy absorption and fracture appearance transition temperature curves for the twelve medium carbon steels. The graphs show the effect of each of four uranium levels at the low sulphur (upper left), medium sulphur (upper right) and high sulphur (lower left) levels.



Effect Of Sulphur On C_v Energy Absorption, At The Nil Uranium Level.



Effect Of Sulphur On C_v Energy Absorption At The 0.14% (Average) Uranium Level.

Figure 12 - Longitudinal C_v energy absorption and fracture appearance transition temperature curves. The graphs show the effect of three levels of sulphur at low and medium carbon levels and at nominal uranium levels of nil (upper left) and 0.14% (upper right).

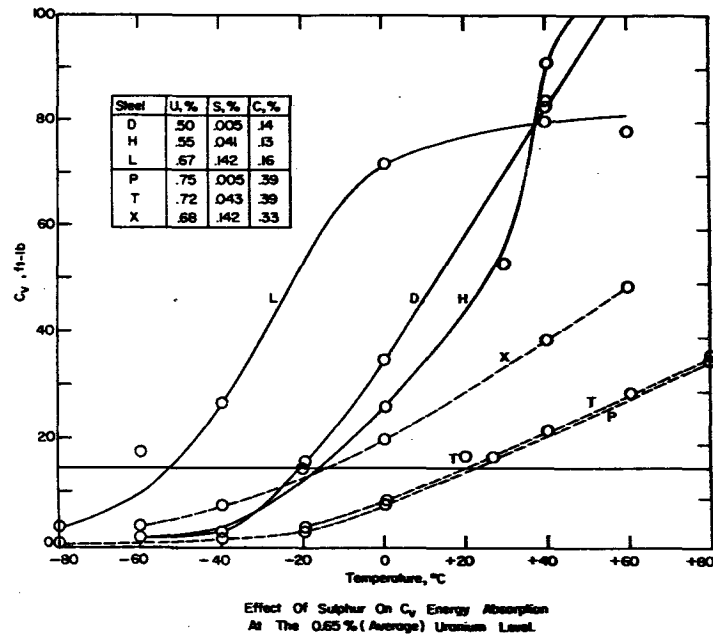
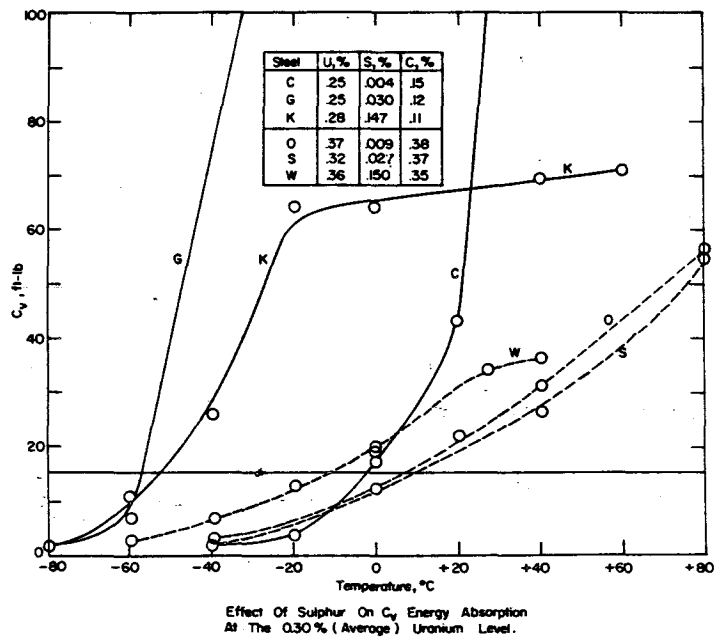


Figure 12 - (continued)

Longitudinal C_v energy absorption and fracture appearance transition temperature curves. The graphs show the effect of three levels of sulphur at low and medium carbon levels and at nominal uranium levels of 0.30% (upper left) and 0.65% (upper right).

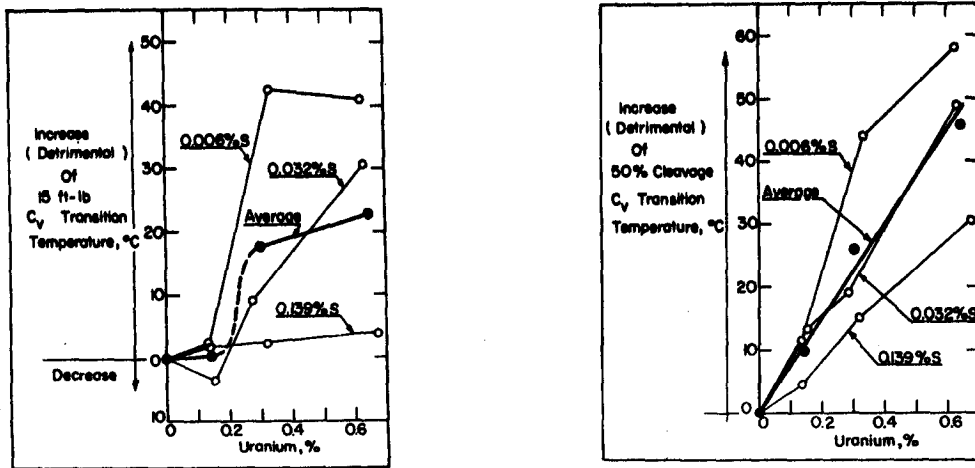


Figure 13 - Graphical representation of the treatment effects of uranium on the C_V 15 ft-lb (left) and 50% cleavage (right) transition temperatures. The overall effects of uranium are highly significant ($\alpha = 0.005$). The effect of uranium on the C_V 15 ft-lb transition temperature also has a significant cubic component ($\alpha = 0.05$). Both graphs indicate that the effect of uranium depends on the sulphur level. Statistically the linear uranium x linear log sulphur interaction components are significant ($\alpha = 0.005$ for C_V 15 ft-lb; $\alpha = 0.10$ for C_V 50% cleavage).

Each open circle value is based on a mean of 2 transition temperatures. Each filled circle value is based on a mean of 6 transition temperatures.

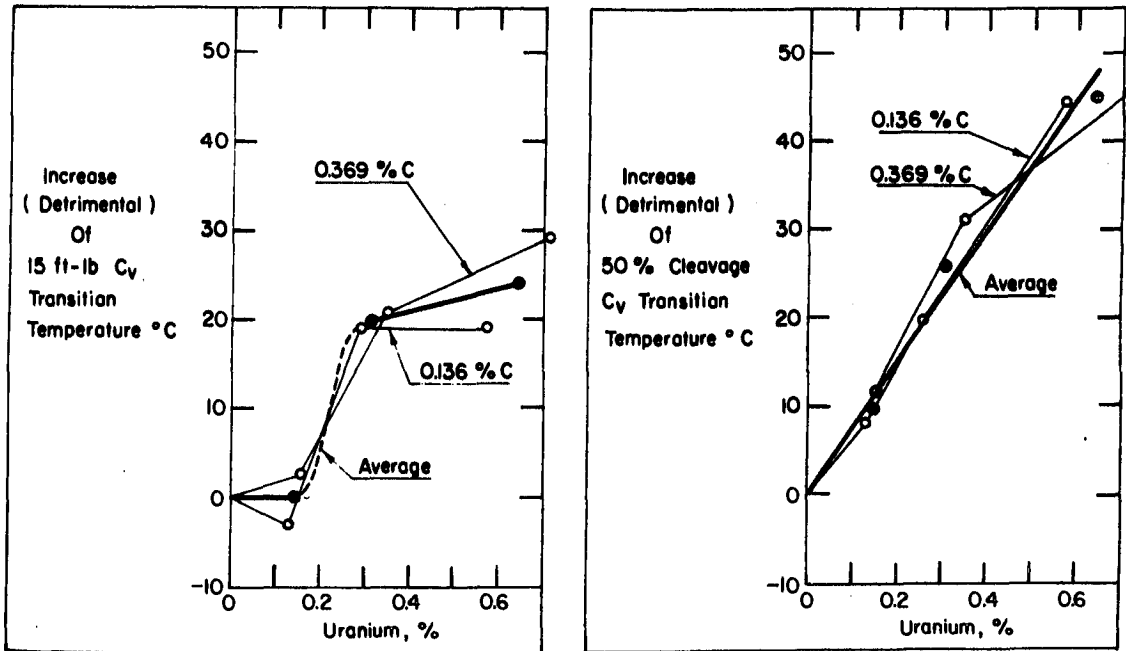


Figure 14 - Graphical representation of the treatment effects of uranium on the C_v 15 ft-lb (left) and 50% cleavage (right) transition temperatures. The overall effect and the effects at each carbon level are shown to be highly significant ($\alpha = 0.005$) with no uranium x carbon interaction.

Each open circle value is based on a mean of 3 transition temperatures. Each filled circle value is based on a mean of 6 transition temperatures.

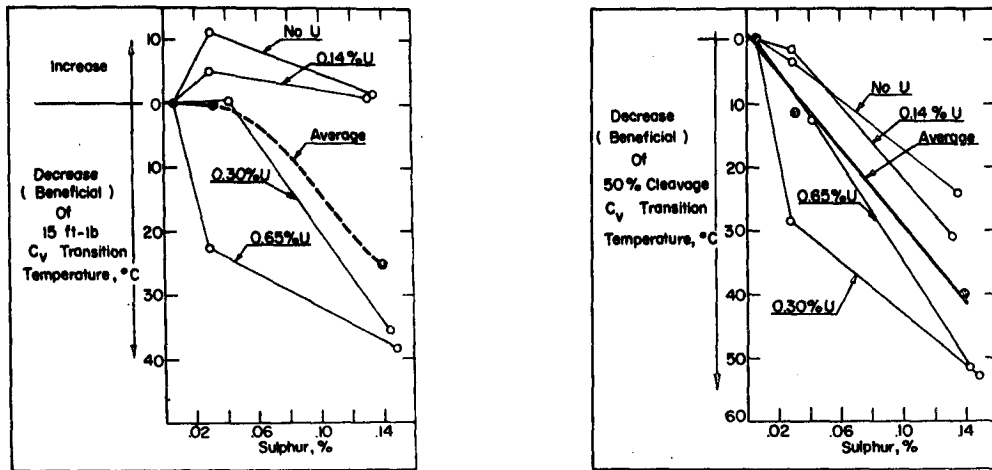


Figure 15 - Graphical representation of the treatment effects of sulphur on the C_v 15 ft-lb (left) and 50% cleavage (right) transition temperatures. The overall effect and the linear component of log sulphur on the C_v 15 ft-lb transition temperature are significant ($\alpha = 0.025$). The overall effect and the linear component of log sulphur on the C_v 50% cleavage transition temperature are highly significant ($\alpha = 0.005$).

Each open circle value is based on a mean of 2 transition temperatures. Each filled circle value is based on a mean of 8 transition temperatures.

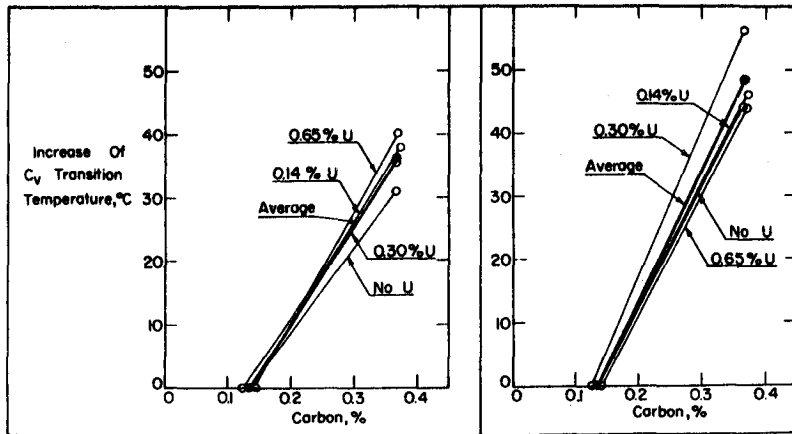


Figure 16 - Graphical representation of the treatment effect of carbon on the C_v 15 ft-lb (left) and 50% cleavage (right) transition temperatures. There is no indication of a carbon x uranium interaction.

Each open circle value is based on a mean of 3 transition temperatures. Each filled circle value is based on a mean of 12 transition temperatures.

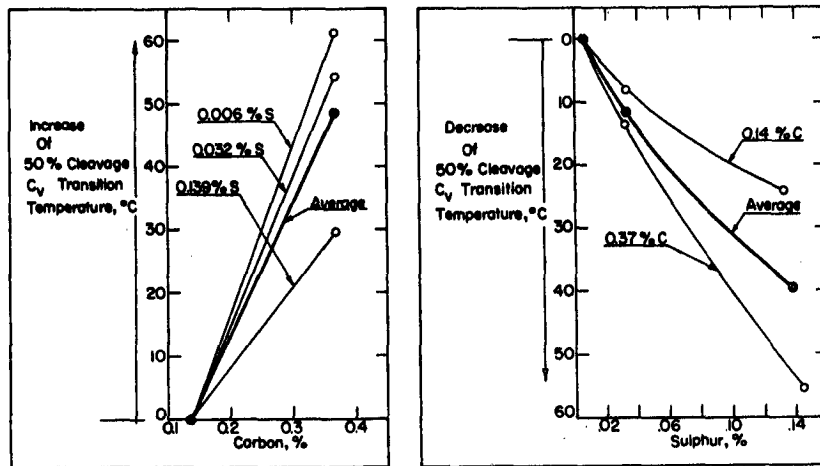


Figure 17 - Graphical representation of the treatment effect of carbon (left) and sulphur (right) on the C_v 50% cleavage transition temperature. The effect of carbon is shown at three sulphur levels. The effect of sulphur is shown at two carbon levels. The apparent sulphur x carbon interaction is not statistically significant with the available experimental data.

Each open circle value is based on a mean of 4 transition temperatures. Each filled circle value is based on a mean of 12 (carbon) or 8 (sulphur) transition temperatures.