Contributions of Selected Residual Elements to the Radiation-Embrittlement Sensitivity of Steel Forgings

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Abstract: Residual impurity element content has a major influence on the sensitivity of low-alloy steels to radiation-induced embrittlement. The identification of phosphorus as highly detrimental to irradiation performance has made tin, arsenic, and antimony additional suspect elements.

The influence of tin, arsenic, and antimony on elevated-temperature radiation-embrittlement sensitivity was explored using a series of seven experimental NiCrMoV forgings. The forgings represented high and low phosphorus contents in addition to statistical variations of tin, arsenic, and antimony. Assessments of relative radiation sensitivity involved the exposure of Charpy-V specimens at 550°F (288°C) to a neutron fluence of 3.1×10^{19} n/cm^2 >1 MeV.

Postirradiation observations indicate that tin, arsenic, and antimony do not contribute directly to radiation-embrittlement sensitivity, either individually or in combination, for the composition range studied. In contrast, phosphorus ($\approx$0.015%P) was observed to have a pronounced detrimental effect on radiation-embrittlement resistance. Of major significance, the contribution of phosphorus to radiation-embrittlement sensitivity was found greatly reduced by a high (100-150 ppm) tin content. The phosphorus-tin interaction appears to be independent of both antimony and molybdenum content for the levels investigated, but may be dependent on a high ($\approx$150 ppm) arsenic content. Points of similarity and nonsimilarity in the influence of phosphorus and tin on radiation-embrittlement and temper-embrittlement behavior are described.

Postirradiation observations confirm that impurity restrictions have significant capability for improving the radiation-embrittlement resistance of low-alloy steel forgings.

INTRODUCTION

The present investigation was undertaken at the request of AEC sponsors to extend knowledge of residual impurity elements critical to the radiation-embrittlement sensitivity of steel (1). The impurity elements tin, arsenic, and antimony were selected for study because of characteristic similarities to phosphorus. Phosphorus, as noted below, has a known detrimental contribution to radiation-embrittlement behavior. A second objective of the investigation was to verify the possibility of producing radiation-resistant forgings via supplemental impurity restrictions. The investigation received the support and generous cooperation of ASTM Committee A-1 on Steel (Subcommittee VI on Forgings).

NRL Problem 63M01-14; Projects RR022-11-41-5409, AEC-AT(49-5)-2110, and MIPR-ERG-41004. This is a final report on one phase of the problem; work on other phases is continuing. Manuscript submitted November 16, 1972.

Pronounced detrimental effects of impurities on radiation-embrittlement resistance have been revealed both with elevated-temperature [≈550°F (288°C)] and with low-temperature [<390°F (199°C)] irradiations, which are representative of water-cooled reactor vessel operations and gas-cooled reactor vessel liner applications, respectively (1,2). By selective restrictions on the content of certain impurity elements, notably copper and phosphorus, very marked improvements to elevated-temperature radiation resistance have been shown possible for low-alloy steel plates (1,3) and for weld fillers (4,5). These improvements have been verified for plates and weld metals in commercial-scale demonstrations (3,5).

The previous determination (1) of a key role of phosphorus in radiation-sensitivity development is shown in Fig. 1. Indirect experimental evidence suggests that the mechanism for the phosphorus contribution to radiation embrittlement is similar to that by which this element contributes to temper embrittlement (6,7). Like phosphorus, the elements tin, arsenic, and antimony have been associated with temper embrittlement (8). It was thus considered that these elements have a potential for influencing radiation-sensitivity level. Although normally present only in small amounts, each of the elements is suspected of having overriding segregation tendencies.

Experimental NiCrMoV steel forgings from the ASTM Temper Embrittlement Program (9) were employed for this study. The forgings were specially selected to represent two levels of tin, arsenic, and antimony content in statistical combinations and, in addition, two levels of phosphorus content. Accordingly, individual element contributions, as well as element-element interactions, could be assessed. It was anticipated that a detrimental role of phosphorus in the irradiation response of forgings would be observed.

The analysis presented herein is developed on the basis of relative pre-postirradiation Charpy-V performance. The findings have major significance not only to the development of radiation-resistant steel forgings but also to an improved understanding of the irradiation behavior of high-strength (>100 ksi yield) steels in general.

MATERIALS

The chemical compositions and heat treatment of the seven forgings selected for study are given in Table 1. The bars (1-1/16 in. square) were obtained from 50-lb vacuum-induction melts. Details of melting procedures and ingot processing are reported elsewhere (9). For ready data comparisons, each forging is assigned a four-place code identifying its particular composition variation with regard to, respectively, phosphorus, tin, arsenic, and antimony content, as follows: high (+), low (−), midrange (0).

Preirradiation mechanical properties of the forgings are given in Table 2. Property differences among the materials appear quite small. Selected microstructures pertinent to the analysis of postirradiation performance are reproduced in Fig. 2; only subtle differences are noted among the structures.

MATERIALS IRRADIATION

The irradiation of Charpy-V specimens was performed in the Union Carbide Research Reactor (UCRR) at 550°F (288°C). All forging codes were irradiated simultaneously to permit

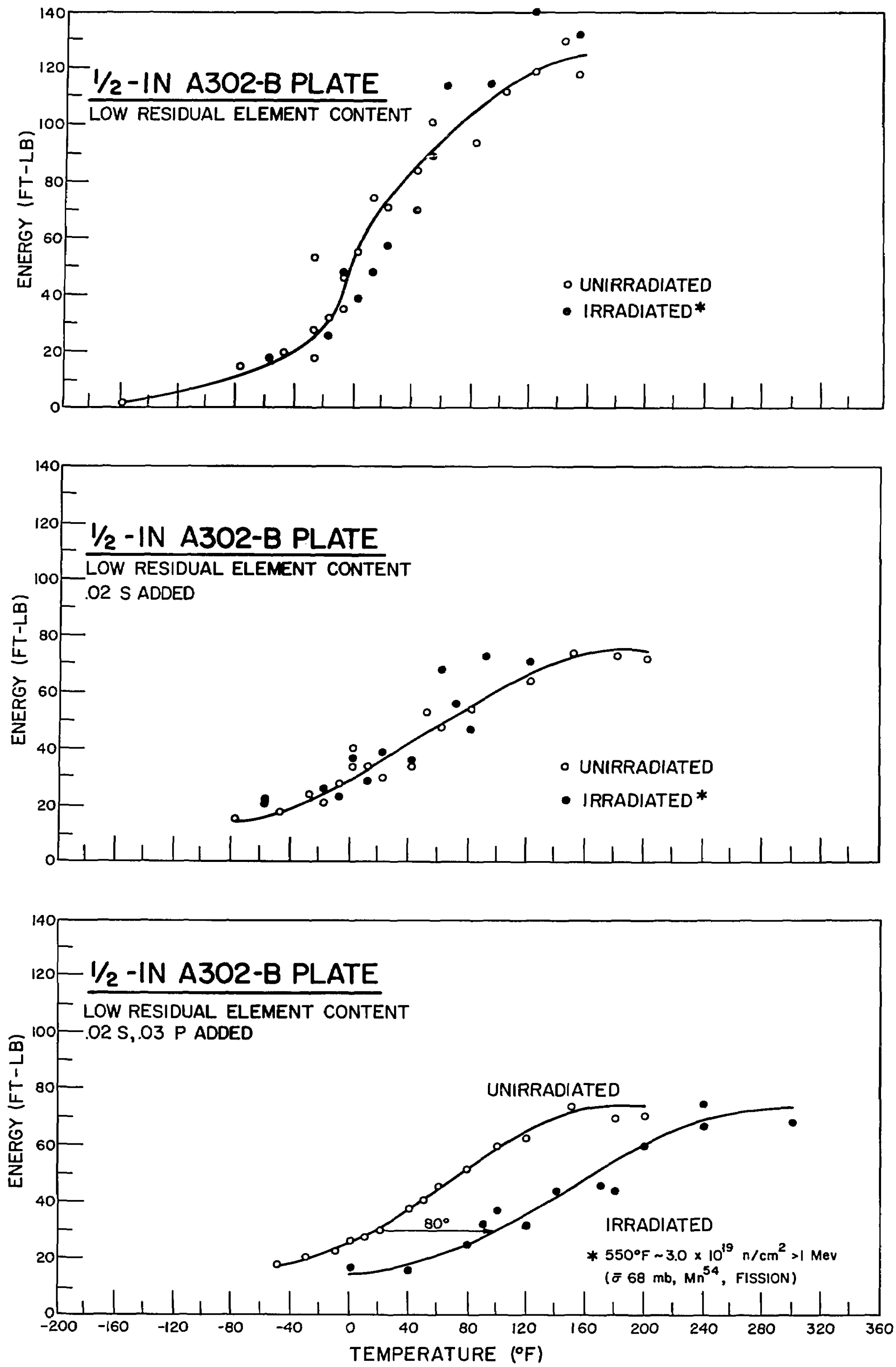

Fig. 1 — Notch ductility behavior of A302-B test plates (sulfur-phosphorus series) before and after irradiation at 550°F (288°C) for 3-way split melt. Top: heat Y9240, cast 1A; middle: heat Y9240, cast 1B; bottom: heat Y9240, cast 1C. (Figure taken from Ref. 1.)

Table 1
Chemical Composition and Heat Treatment of the NiCrMoV Forgings. (Data from Ref. 9.)

Melt/Forging	Code†	Chemical Composition* (wt-% unless otherwise noted)														
		P	Sn (ppm)	As (ppm)	Sb (ppm)	Mo	C	Mn	Si	S	Ni	Cr	V	Cu‡	N (ppm)	Al
																soluble	total
P	−−−0	0.006	15	19	18	0.31	0.24	0.31	0.056	0.008	3.47	1.72	0.12	0.006	21	0.010	0.016
V	−−++	0.006	19	160	27	0.30	0.25	0.32	0.044	0.011	3.45	1.74	0.11	0.006	29	0.009	0.013
O	−+++	0.006	105	140	30	0.59	0.27	0.31	0.062	0.009	3.48	1.71	0.12	—	25	0.008	0.012
J	−+−+	0.003	91	36	27	0.29	0.26	0.31	0.059	0.007	3.46	1.72	0.11	0.008	22	0.004	0.009
C	++++	0.019	137	170	30	0.31	0.24	0.31	0.047	0.011	3.57	1.76	0.12	0.010	31	0.002	0.004
G	++−+	0.016	20	150	30	0.60	0.23	0.30	0.057	0.008	3.52	1.75	0.12	0.008	27	0.010	0.012
B	+++−	0.018	98	150	10	0.61	0.24	0.31	0.053	0.009	3.54	1.74	0.12	0.006	30	0.002	0.003

*Courtesy ASTM Subcommittee VI on Forgings (Subgroup on Temper Embrittlement).
†Composition variation of P, Sn, As, and Sb, respectively: high (+), low (−), midrange (0).
‡NRL determination.

Heat Treatment

Austenitized by rapid heating to 1200°F (649°C), heating from 1200°F (649°C) to 1550°F (843°C) at 50°F/hr (28°C/hr), holding at 1550°F (843°C) for 38 hr, cooling to room temperature following curve for center of 54-in.-diam water-quenched rotor.

Tempered at 1110-1130°F (599-610°C) for 48 hr, air cooled; retempered at 1090-1110°F (588-599°C) for 48 hr, air cooled.

Table 2
Preirradiation Mechanical Properties* of the NiCrMoV Forgings

Melt/Forging	Code†	Yield Strength (0.2% offset) (ksi)	Tensile Strength (ksi)	Elongation (%)	Reduction in Area (%)	Charpy-V 30 ft-lb Temperature		Charpy-V FAT_{50} Temperature‡	
						(°F)	(°C)	(°F)	(°C)
P	−−−0	106.0	123.0	20.5	69.0	−130	−90	−65	−54
V	−−++	107.0	125.0	20.5	69.0	−135	−93	−85	−65
O	−+++	108.0¶	126.0	22.0	71.4	−140	−96	−90	−68
J	−+−+	105.0	123.0	21.0	70.8	−155	−104	−90	−68
C	++++	106.0	123.5	20.5	69.9	−140	−96	−85	−65
G	+−++	105.0	123.5	21.5	69.9	−125	−87	−55	−48
B	+++−	105.5	124.5	21.0	68.6	−115	−82	−60	−51

*Courtesy ASTM Subcommittee VI on Forgings (Subgroup on Temper Embrittlement).
†Composition variation, etc.—see Table 1.
‡Fracture Appearance Transition (50% cleavage or intergranular fracture).
¶0.252-in.-diam specimen.

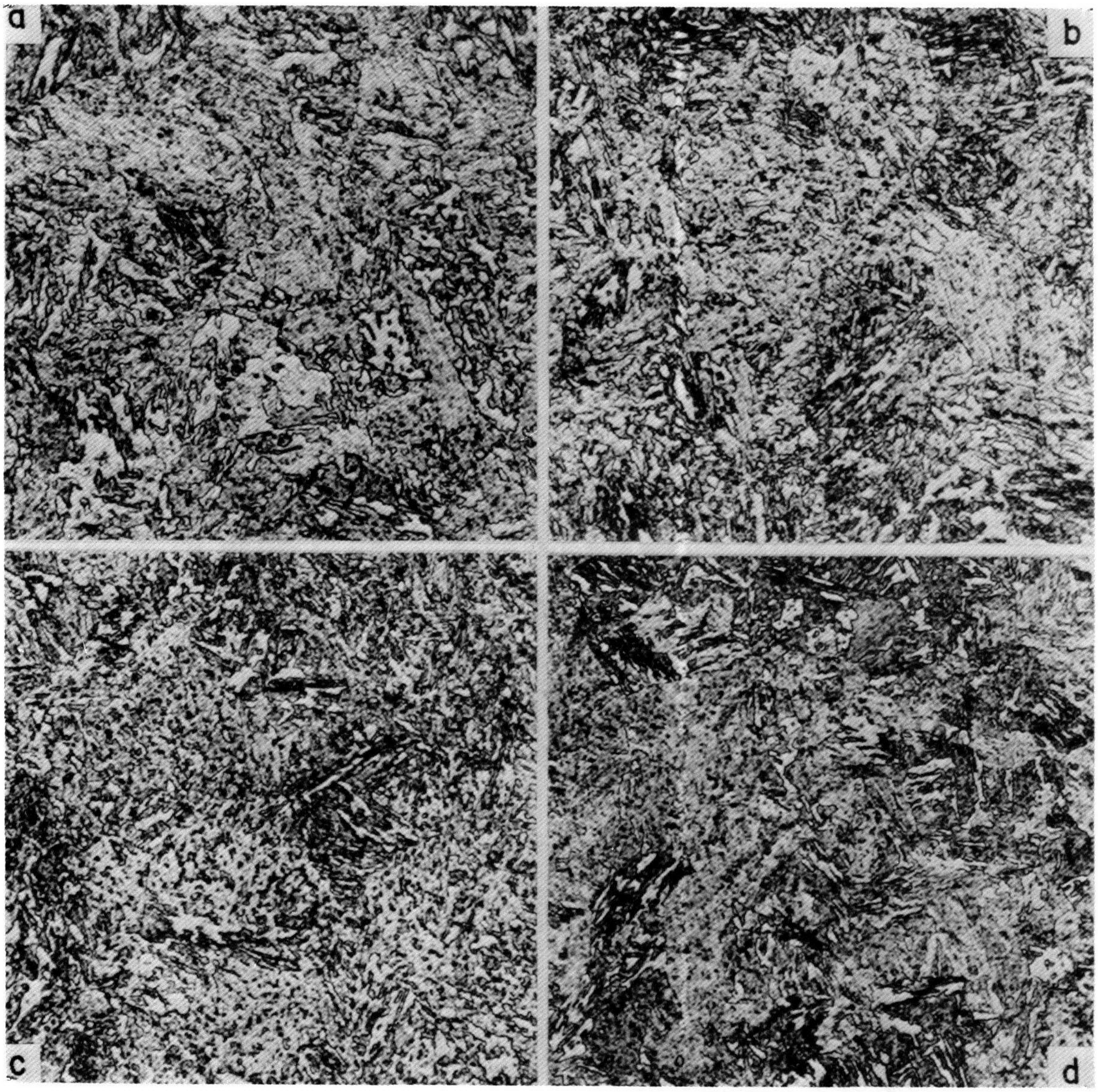

Fig. 2 – Microstructures of forgings P, C, G, and B (tempered bainite, 400X).

direct comparisons of irradiation response characteristics. During irradiation, specimen temperatures were monitored continuously by thermocouples. The neutron fluence received during the 1000-hr exposure was 3.1×10^{19} n/cm^2 >1 MeV as determined from iron neutron dosimeter wires ($^{54}Fe(n,p)^{54}Mn$ reaction) included in the specimen array.

RESULTS

Experimental results are presented in Figs. 3 and 4 and are summarized in Table 3. The graphs are grouped according to phosphorus content (low, Fig. 3, and high, Fig. 4). Data representing 550°F (288°C) 1000-hr thermal control tests are included in each graph. Significant changes in Charpy-V or tensile properties were not induced by the thermal conditioning treatment. The most apparent change (Table 4) was a small (10 to 15 ft-lb) elevation in Charpy-V upper shelf energy.

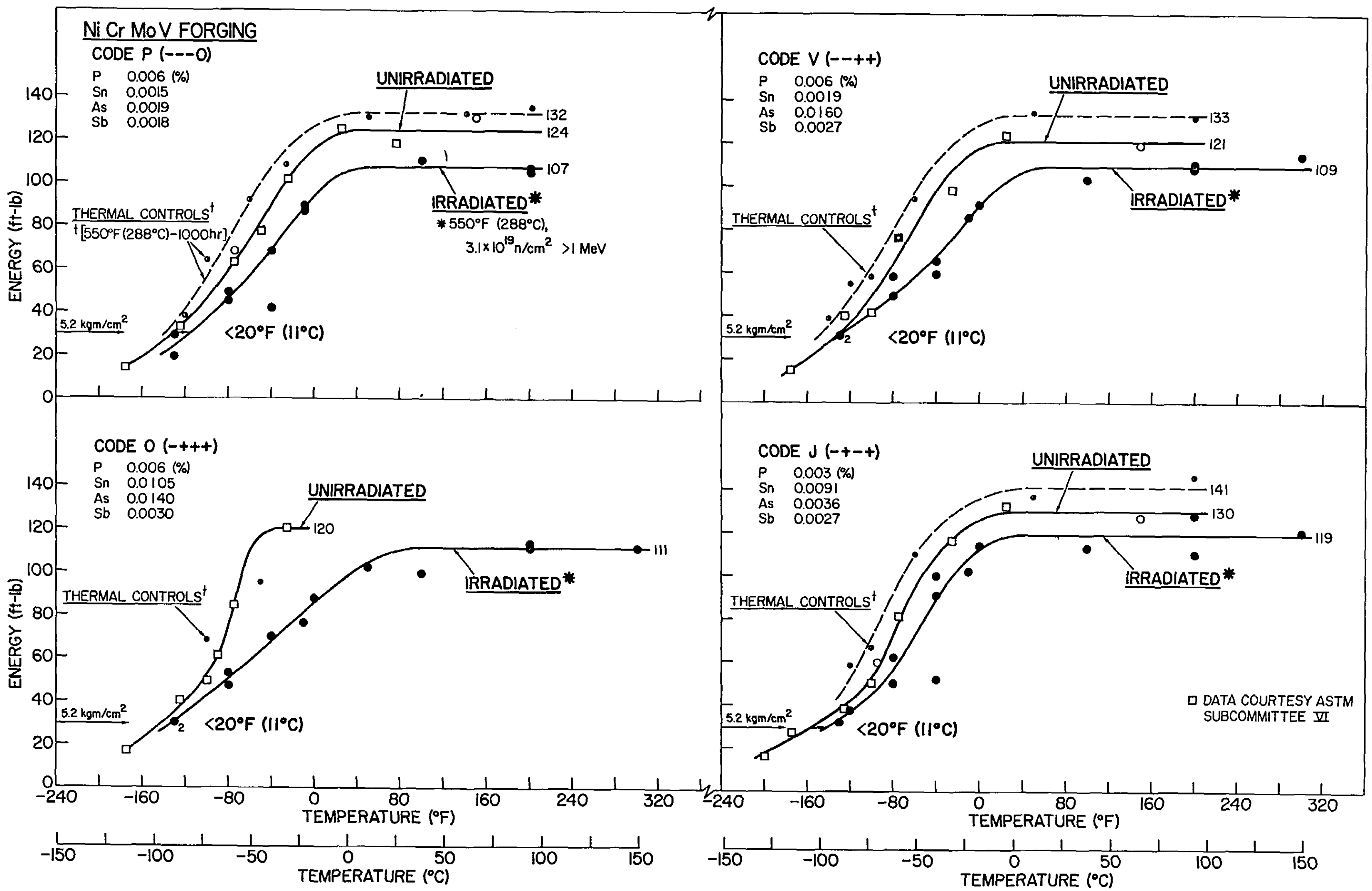

Fig. 3 – Postirradiation Charpy-V performance of forgings P, V, O, and J representing the low-phosphorus-content group. Results for thermal conditioning (550°F (288°C), 1000 hr) of unirradiated specimens are also shown.

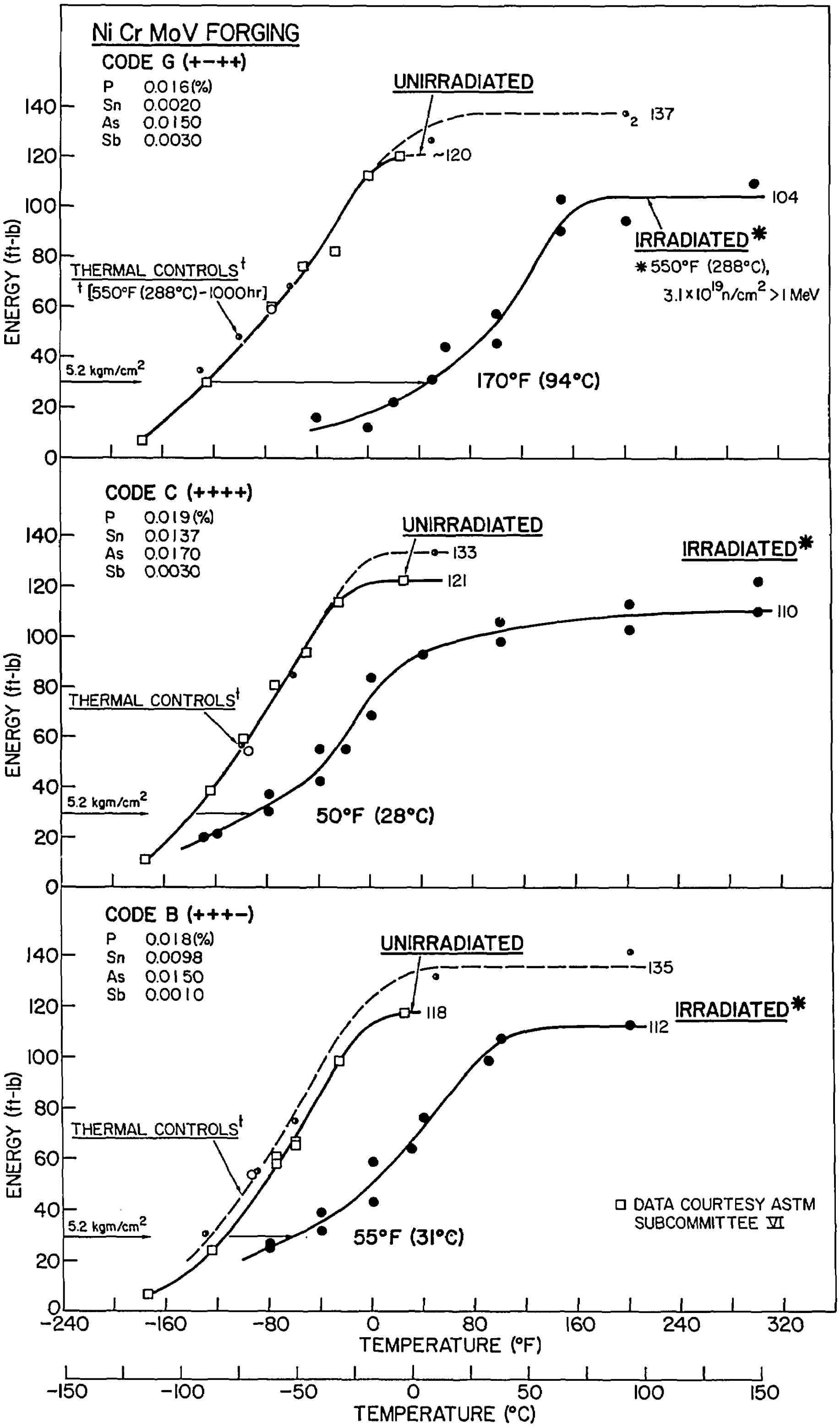

Fig. 4 – Postirradiation Charpy-V performance of forgings G, C, and B representing the high-phosphorus-content group. Results for thermal conditioning (550°F (288°C), 1000 hr) of unirradiated specimens are also shown.

Table 3
Postirradiation Charpy-V Notch Ductility of the NiCrMoV Forgings

Melt/Forging	Code*	Charpy-V 30-ft-lb Transition						Charpy-V 60-ft-lb Transition						Charpy-V Shelf Energy		
		Initial		Final		Increase		Initial		Final		Increase		Initial	Final	Decrease
		(°F)	(°C)	(°F)	(°C)	Δ(°F)	Δ(°C)	(°F)	(°C)	(°F)	(°C)	Δ(°F)	Δ(°C)	(ft-lb)	(ft-lb)	Δ(ft-lb)
P	−−−0	−130	−90	−115	−82	15	8	−80	−62	−55	−48	25	14	≈124	107	≈17
V	−−++	−135	−93	−130	−90	5	3	−85	−65	−55	−48	30	17	121	109	12
O	−+++	−140	−96	−130	−90	10	6	−90	−68	−55	−48	35	20	≈120	111	≈9
J	−+−+	−155	−104	−140	−96	15	8	−90	−68	−75	−59	15	9	130	119	11
C	++++	−140	−96	−90	−68	50	28	−95	−71	−20	−29	75	42	121	110	11
G	+−++	−125	−87	45	7	170	94	−75	−59	110	43	185	102	≈120	104	≈16
B	+++−	−115	−82	−60	−51	55	31	−70	−57	20	−7	90	50	≈118	112	≈6

*Composition variation of P, Sn, As, and Sb, respectively: high (+), low (−), midrange (0).

Table 4
Effect of 550°F (288°C) Thermal Conditioning of the NiCrMoV Forgings
(1000-hr Control Tests)

Charpy-V Notch Ductility

Melt/Forging	Code*	Charpy-V 30-ft-lb Temperature				Charpy-V Shelf Energy	
		Final		Decrease		Final	Increase
		(°F)	(°C)	Δ(°F)	Δ(°C)	(ft-lb)	Δ(ft-lb)
P	−−−0	−140	−96	10	6	132	8
V	−−++	−150	−101	15	<11	133	12
O	−+++	−140†	−96†	≤20†	≤11†	—	—
J	−+−+	−155†	−104†	20†	11†	141	11
C	++++	−140	−96	0	0	133	12
G	+−++	−125	−87	0	0	137	≤17‡
B	+++−	−125	−87	10	6	135	≤17‡

Tensile Properties

Melt/Forging	Code*	Yield Strength¶ (0.2% offset, ksi)		Tensile Strength (ksi)		Elongation (%)		Reduction in Area (%)	
		Final	Change	Final	Change	Final	Change	Final	Change
P	−−−0	105.2	−0.8	120.8	−2.2	21.5	+1.0	70.2	+1.2
V	−−++	104.6	−2.4	121.0	−4.0	21.5	+1.0	69.7	+0.7
O	−+++	—	—	—	—	—	—	—	—
J	−+−+	104.6	−0.4	120.8	−2.2	21.5	+0.5	70.6	+0.2
C	++++	105.8	−0.2	121.3	−2.2	21.5	+1.0	70.2	+0.3
G	+−++	104.6	−0.4	120.7	−2.8	21.5	0	70.0	+0.1
B	+++−	104.8	−0.7	121.0	−3.5	21.5	+0.5	70.5	+1.9

*Composition variation of P, Sn, As, and Sb, respectively: high (+), low (−), midrange (0).
†Estimate based on 60-ft-lb transition behavior.
‡Initial shelf energy value not fully established.
¶0.505-in.-diam tensile specimen; single tests.

In Fig. 3, showing the performance of the low-phosphorus-content forgings, it is evident that the variations in tin, arsenic, and antimony did not influence the irradiation response characteristics of this group. In each case, the increase in the Charpy-V 30 ft-lb (5.2 kg-m/cm^2) transition temperature* by irradiation was less than 20°F (11°C) and the radiation-induced decrease in the upper shelf level was on the order of 10 ft-lb. Accordingly, it would appear that tin, arsenic, and antimony do not have a direct contribution to radiation-embrittlement

*The Charpy-V 30 ft-lb (5.2 kg-m/cm^2) temperature is often used as a convenient arbitrary index of brittle/ductile transition for pre-postirradiation comparisons of steel performance.

sensitivity, either individually or in combination, for the content range investigated. The same conclusion is reached if a higher Charpy-V index (60 ft-lb) is chosen to compare pre-postirradiation performance.

Figure 4 contrasts the results for the high-phosphorus-content group. The sensitizing effect of phosphorus content noted with Fig. 1 is clearly evident from the individual transition temperature responses. Of greater significance, data comparisons within this group suggest that tin has the capacity to decrease markedly the detrimental effect of a high phosphorus content. Specifically, the transition temperature increases for forgings B and C were only one third of that for forging G. Assuming that future tests confirm this indication of a phosphorus-tin interaction, the results will mark one of few cases noted of one element countering another element in terms of radiation-sensitivity development in low-alloy steels.

Additional intercomparisons from Fig. 4 suggest that the benefit of tin content is independent of the levels of antimony and molybdenum for the ranges investigated. It is possible, however, that the benefit derived from a higher tin content is in itself dependent on having the higher arsenic content. The test matrix was insufficient to clarify or confirm a synergism in this case. Similarly, the phosphorus-tin interaction may partially depend on a moderately high steel yield strength (>100 ksi yield).

A final observation which concerns the second objective of the investigation is the very high [550°F (288°C)] radiation-embrittlement resistance exhibited by all four low-phosphorus-content forgings, (Fig. 3). For unimproved reactor vessel steels, including A302-B and A533-B plates and weld deposits, transition temperature increases on the order of 150-225°F (83-125°C) are normally anticipated for the irradiation conditions of this investigation (3). From this contrast, it is clear that the 550°F (288°C) radiation resistance of forging materials can be enhanced through proper restrictions on impurity element content.

DISCUSSION

The observation that phosphorus and tin have a major influence on radiation-embrittlement sensitivity is consistent with observations that these same elements are critical to temper-embrittlement tendencies. An apparent interaction between the two elements is also indicated in each case. However, important differences are noted. First, the interaction between phosphorus and tin provides reduced radiation-embrittlement sensitivity, whereas the interaction observed in temper embrittlement is toward enhanced embrittlement formation. Second, tin does not appear to make a direct contribution to radiation-embrittlement sensitivity but does show a direct contribution to temper-embrittlement tendencies. Thus, the contributions of phosphorus and tin to radiation-embrittlement and temper-embrittlement behavior are similar in character in some but not all respects. To cite other parallel observations, antimony and arsenic were not found to contribute directly to the radiation- or temper-embrittlement behavior of the forgings in the described heat treatment condition. The lack of an effect from antimony, however, may be due to its low content in the materials. A possible synergism between arsenic and tin is also noted.

It is known that copper, in addition to phosphorus, has a major detrimental influence on radiation-embrittlement resistance (1) and that the two elements have additive sensitizing influences (4). It has been determined further that the copper contribution takes place through an enhancement of the yield strength elevation by irradiation (6,7) and that the mechanism, at the microscale, is a heterogeneous nucleation of vacancy defects by copper atoms, or small atom clusters (7). Parallel evaluations of the phosphorus contribution, however, have revealed neither an influence on yield strength behavior nor an influence on defect nucleation (7). Accordingly, it must be presumed that the mechanism for the phosphorus contribution is significantly different from that identified for copper.

It was proposed earlier that the means by which phosphorus enhances apparent radiation embrittlement is through a combination of radiation-enhanced diffusion and the weakening of ferrite-carbide interfaces (6). This would be similar to its mechanism for temper-embrittlement formation where prior austenite grain boundaries are involved. To explain the current findings within the framework of this proposal, it would be necessary for either the diffusivity of phosphorus during irradiation to be reduced by the presence of tin, or that tin be moved in preference to phosphorus to the interfaces, with a subsequent reduction in embrittlement severity. Auger spectroscopy may clarify which mechanisms are involved in the phosphorus contribution and in the phosphorus-tin interaction. Exploratory studies with samples from the melt of Fig. 1 (casts 1B and 1C) are underway.

A further understanding of the conditions under which tin contributes to radiation resistance may explain some anomalies noted in past irradiation studies. Unfortunately, composition documentation for reactor structural steels generally has not included tin content determinations. (Until recently a comparable situation existed with regard to copper content.) To preclude this type of situation, it is highly recommended that well-documented archive material be reserved from critical reactor components whenever possible.

CONCLUSIONS

The primary observations and conclusions of this study concerning 550°F (288°C) radiation-embrittlement resistance are as follows:

1. Tin, arsenic, and antimony do not contribute directly to radiation-embrittlement sensitivity, either individually or in combination, for the composition range investigated. All four forgings having a low phosphorus content (0.006%P max) were highly radiation resistant, irrespective of tin, arsenic, or antimony content level.

2. A high phosphorus content ($\approx$0.015%P) has a pronounced detrimental effect on the radiation-embrittlement resistance of low-alloy steel forgings. This determination is consistent with prior determinations of a detrimental effect of phorphorus content on the radiation resistance of low-alloy steel plates and weld deposits.

3. Tin content can reduce the contribution of phosphorus content to the radiation-embrittlement sensitivity of low-alloy steel forgings very markedly. The benefit of a high tin content ($\approx$100 ppm Sn) was found to be independent of antimony content and molybdenum content for the ranges investigated, but the benefit is possibly dependent on a high arsenic content ($\approx$150 ppm As) and a moderately high yield strength ($\approx$100 ksi yield).

4. Radiation embrittlement and temper embrittlement are similar in that both are dependent on phosphorus and tin content and both reflect phosphorus-tin interactions. However, phosphorus-tin interactions are beneficial with regard to radiation-embrittlement resistance but are detrimental to temper-embrittlement resistance.

5. Restrictions on impurity element content can result in a significant improvement to the radiation-embrittlement resistance of low-alloy steel forgings, similar to the improvement demonstrated for low-alloy steel plates and weld metals.

It is emphasized that the findings of the present investigation represent only a limited effort toward understanding the contributions of tin, arsenic, and antimony to radiation-embrittlement sensitivity. Several factors could have a potential bearing on the individual and combined effects of these elements, including base alloy composition, deoxidation practice, yield strength level, grain size, and heat treatment condition. Clarifying investigations to explore the significance of these variables are considered desirable.

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13. ABSTRACT

Residual impurity element content has a major influence on the sensitivity of low-alloy steels to radiation-induced embrittlement. The identification of phosphorus as highly detrimental to irradiation performance has made tin, arsenic, and antimony additional suspect elements.

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Postirradiation observations indicate that tin, arsenic, and antimony do not contribute directly to radiation-embrittlement sensitivity, either individually or in combination, for the composition range studied. In contrast, phosphorus ($\approx$0.015%P) was observed to have a pronounced detrimental effect on radiation-embrittlement resistance. Of major significance, the contribution of phosphorus to radiation-embrittlement sensitivity was found greatly reduced by a high (100-150 ppm) tin content. The phosphorus-tin interaction appears to be independent of both antimony and molybdenum content for the levels investigated, but may be dependent on a high ($\approx$150 ppm) arsenic content. Points of similarity and nonsimilarity in the influence of phosphorus and tin on radiation-embrittlement and temper-embrittlement behavior are described.

Postirradiation observations confirm that impurity restrictions have significant capability for improving the radiation-embrittlement resistance of low-alloy steel forgings.

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